

The Journal of the Society of Dyers and Colourists

Volume 76



Number 7

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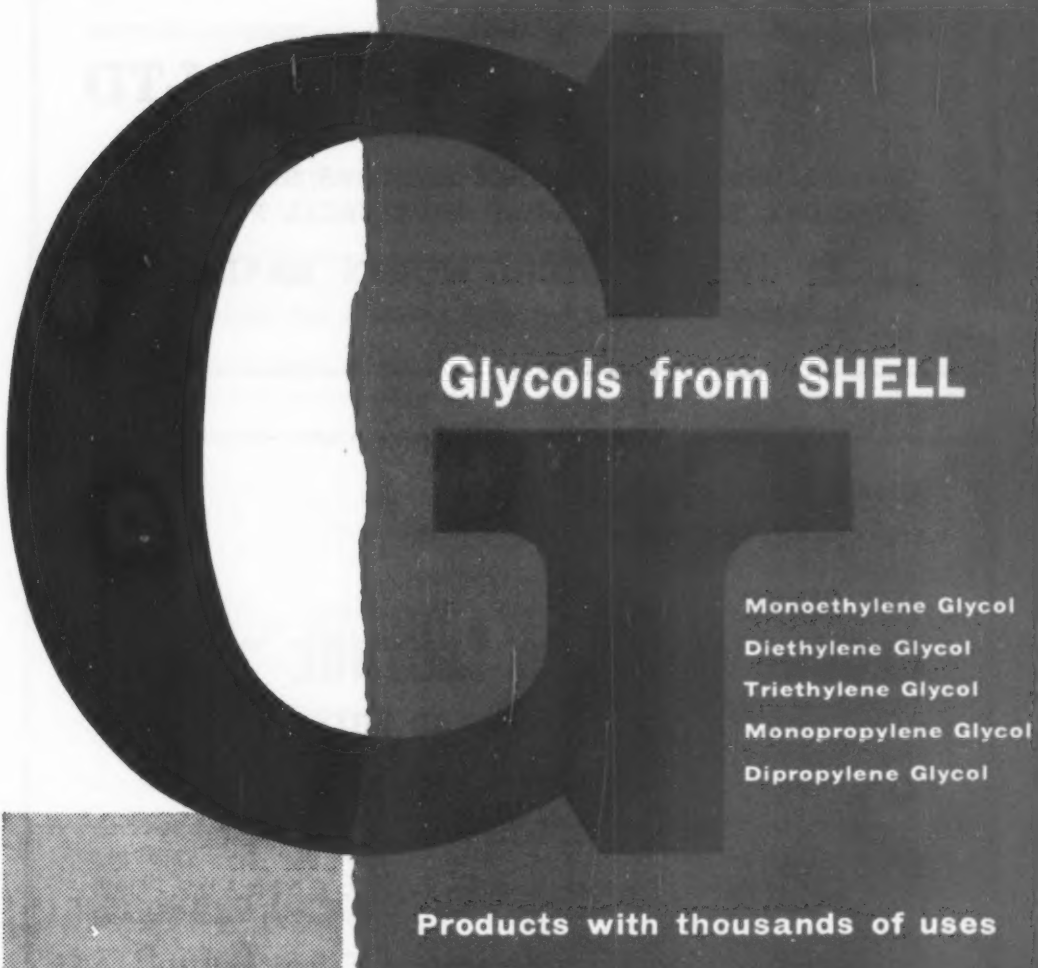
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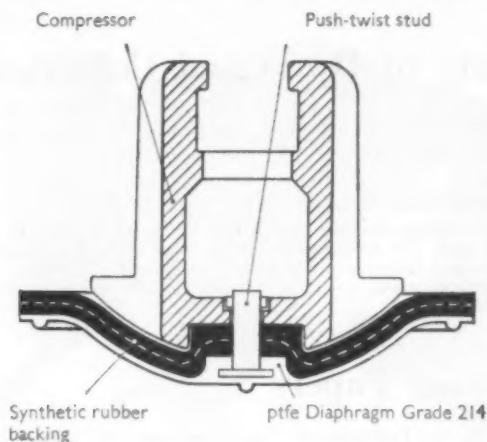
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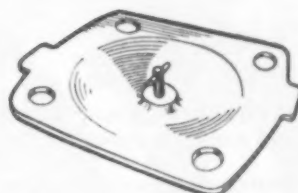


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Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Use of Colour in Cosmetics	<i>D. F. Anstead</i>
The Paper Chromatography of Synthetic Dyes	<i>J. C. Brown</i>
Antistatic Agents in the Textile Industry	<i>A. E. Henshall</i>
The Theory and Practice of Sodium Chlorite Bleaching	<i>J. K. Skelly</i>

COMMUNICATIONS

The Effect of Environment and Substituents on the Photochemical Activity of Anthraquinonoid Vat Dyes and the Role of $n \rightarrow \pi^*$ Transitions	<i>N. K. Bridge</i>
Relation of Apparent Diffusion Coefficient to the Time of Half-dyeing	<i>W. F. Kilby</i>
Dyeing Education in Glasgow	<i>R. A. Peel</i>

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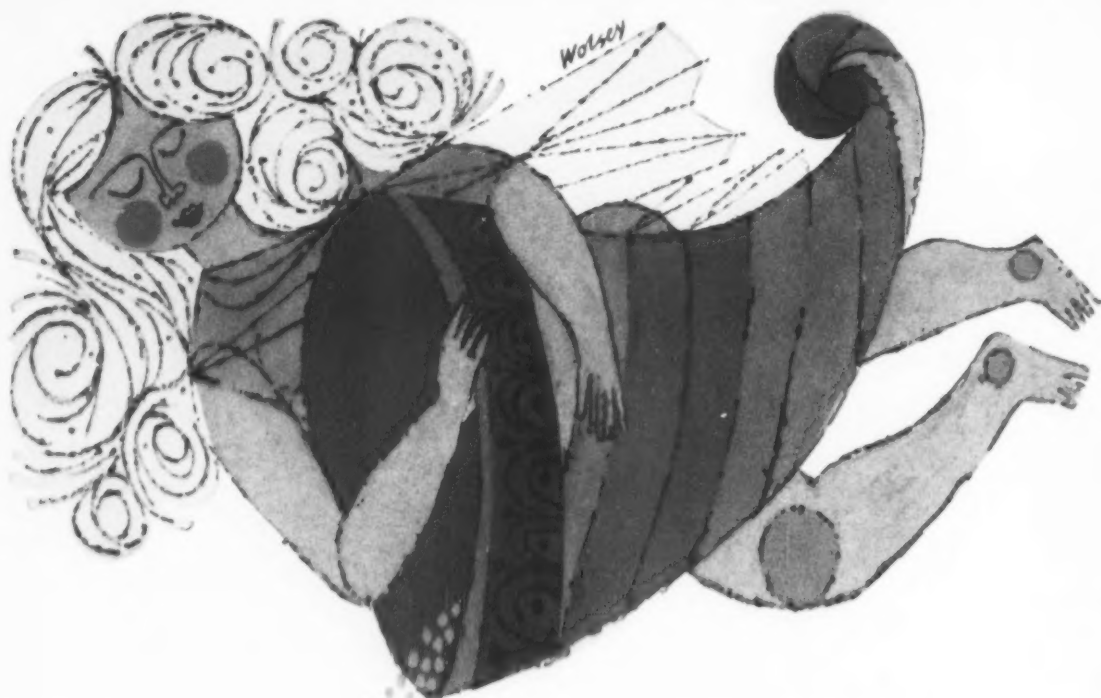
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(* ex officio member)

Honorary Secretary ESMÉE SMITH (Miss), Flat 16, Foxhill, Weetwood Lane, Leeds 16

BRADFORD JUNIOR BRANCH

Chairman E. N. WOOD

Committee

I. Carter	T. Hardisty	R. P. Meredith	T. M. Rodgers
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Honorary Secretary W. R. HERMES Jnr., 69 Manston Way, Crossgates, Leeds 15

LEEDS JUNIOR BRANCH

Chairman J. M. WYLES Vice-chairman M. FRANKS

Committee

D. Balmforth	J. D. Jennison	J. H. Pierce	R. Seddon
D. Batty			

Honorary Secretary J. ELLIOTT

Department of Colour Chemistry and Dyeing, The University, Leeds 2

MANCHESTER JUNIOR BRANCH

Chairman A. S. FERN Vice-chairman H. A. TURNER

Committee

R. D. Ellor	W. S. Hickman	D. P. Oulton	F. Porter
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Honorary Secretary W. R. LEIGH, 1 Grange Road, Bromley Cross, Bolton, Lancs.

SCOTTISH JUNIOR BRANCH

Chairman W. A. BLUE Vice-chairman G. H. MATTINSON

Committee

*F. Attack	*I. Gailey	*J. G. B. McCallum	J. J. H. Storm
*W. P. Edmondson	A. M. Love	C. McNeil	

(* ex officio member)

Honorary Secretary R. McDONALD, Westlea, 24 Methuen Road, Paisley, Scotland

AFFILIATED SOCIETIES

THE SOCIETY OF DYERS AND COLOURISTS OF AUSTRALIA

<i>President</i>	E. I. Noble
<i>Immediate Past President</i>	W. H. Wainwright
<i>Honorary General Treasurer</i>	W. C. Castle
<i>Honorary General Secretary</i>	J. Brear c/o ICIANZ Ltd. 529 Lonsdale Street Melbourne
<i>Publications Editor</i>	R. S. Mayston

Section Committees

VICTORIAN

<i>Chairman</i>	R. Brereton
<i>Vice-chairman</i>	J. D. Harkness
<i>Honorary Secretary</i>	P. Law
<i>Assistant Honorary Secretary</i>	G. I. Fergusson
<i>Honorary Treasurer</i>	B. Firth
<i>Counsellor</i>	T. H. Hall
<i>Honorary Librarian</i>	R. S. Mayston
<i>Honorary Social Secretary</i>	A. Munro
<i>Committee</i>	L. Crawshaw, B. R. Knight, A. Morrison, and A. Munro

GEE LONG (SUBSECTION)

<i>Chairman</i>	R. B. Sweetten
<i>Honorary Secretary</i>	E. C. Foster
<i>Honorary Treasurer</i>	Miss L. Hill

SOUTH AUSTRALIAN

<i>Chairman</i>	S. G. Pike
<i>Vice-chairman</i>	J. Allen
<i>Immediate Past Chairman</i>	E. Voitkuns
<i>Honorary Secretary</i>	R. P. Crook
<i>Assistant Honorary Secretary</i>	H. D. Bruce
<i>Honorary Treasurer</i>	I. Emslie
<i>Honorary Librarian</i>	H. D. Bruce
<i>Committee</i>	J. Burns, J. Davids, W. H. Wainwright, L. Way, and G. Wood

NEW SOUTH WALES

<i>Chairman</i>	W. F. Rogers
<i>Vice-chairman</i>	L. L. White
<i>Immediate Past Chairman</i>	H. W. Senior
<i>Honorary Secretary</i>	A. McLean
<i>Assistant Honorary Secretary</i>	J. C. Hardy
<i>Honorary Treasurer</i>	F. Scully
<i>Counsellor</i>	J. J. Eastwood
<i>Honorary Librarian</i>	F. Eastwood

QUEENSLAND

<i>Chairman</i>	J. Robb
<i>Vice-chairman</i>	M. G. Tune
<i>Honorary Secretary</i>	J. Ogilvie
<i>Assistant Honorary Secretary</i>	R. Lambert
<i>Honorary Treasurer</i>	P. Hogg
<i>Committee</i>	I. Curtis, B. Kiernan, P. McCaghern, and H. Tearle

WEST AUSTRALIAN

<i>Chairman</i>	W. Donovan
<i>Vice-chairman</i>	C. J. Cocks
<i>Immediate Past Chairman</i>	J. Smith
<i>Honorary Secretary and Treasurer</i>	C. W. Cruickshank
<i>Committee</i>	J. R. Edinger, H. Knight, D. Lewis, P. G. Murphy, and N. Parker

NEW ZEALAND

<i>Chairman</i>	A. C. Miller
<i>Vice-chairman</i>	G. Bevins
<i>Honorary Secretary</i>	C. J. Ward
<i>Assistant Honorary Secretary</i>	A. G. Stubbs
<i>Honorary Treasurer</i>	W. Mayer

DYERS AND COLOURISTS ASSOCIATION OF SOUTH AFRICA

P.O. Box 930, Durban

<i>Chairman</i>	J. A. Allen
<i>Vice-chairman</i>	E. Hipperson
<i>Secretary</i>	G. H. Hayward
<i>Treasurer</i>	E. Cullis
<i>Committee</i>	E. Cornah and B. Farmer

Retiring President's Message

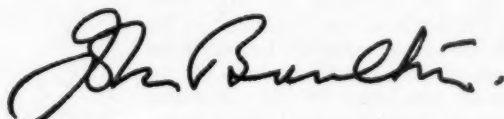
TWO years in office as your President has been an exhilarating experience. The common reward of a retiring President is to look back at such progress as may have been achieved during his term, and take comfort, as he lays down office, from the results of whatever part he has been privileged to play. In this spirit I wish to record my satisfaction at a number of highlights during my term of office; at the same time I wish to offer my warmest thanks to all those in the Society, including the Honorary Officers, members of Council, Chairmen and members of Committees, whose work has made possible what has been done in the last two years. I must mention here also the very great help I have received from the General Secretary and his staff.

I think everyone who attended the International London Congress will agree that this was indeed a highlight. I personally am deeply conscious of the honour that fell to me of acting at the Congress as President both of the Society and of the International Federation of Associations of Textile Chemists and Colourists.

The success of the London Conference is a reflection not only of the Society's standing in the International field, but of the interest which has developed, throughout our membership, in the work and viewpoints of our colleagues in all parts of the world. I know that Council is with me in regarding this Conference as a further step in establishing the very high standing of our Society and our *Journal* all over the world and in contributing to the spirit of friendly co-operation between countries. This co-operation should be actively extended throughout the world, wherever colourists and textile chemists are at work.

Another landmark has been the establishment during the last year of a new section of our Society at home. I refer to the West of England and South Wales Section, which has got off to a very good start and to members of which I send a special message of goodwill for their future.

Working at the heart of the Society's activities I have seen to what extent the Society's standing and health stems directly from the unselfish work of the individuals who make up our membership, and I realise afresh how fortunate we are in goodwill between our Society and the industry and the seats of learning which are in our field. In closing this message I would express my gratitude for all the personal kindness and help I have been shown by everyone in the Society with whom I have come in contact during my term of office: they have made a pleasure of the hard work we have shared.



11th March 1960

Proceedings of the Society

Annual General Meeting

The Seventy-sixth Annual General Meeting of the Society of Dyers and Colourists was held at the Queen's Hotel, Leeds, on Friday, 29th April 1960, at 3.45 p.m., the President (Mr. JOHN BOULTON) being in the chair, and 112 members of the Society being present.

The President extended a welcome to all present.

1. NOTICE CONVENING THE MEETING

The General Secretary read the Notice convening the Meeting.

2. MINUTES OF THE SEVENTY-FIFTH ANNUAL GENERAL MEETING HELD ON 10TH APRIL 1959

Mr. H. A. TURNER proposed that the Minutes as circulated be adopted, Mr. S. R. COCKETT seconded, and it was unanimously—

Resolved that the Minutes of the Seventy-fifth Annual General Meeting of Members held on 10th April 1959, at the Grand Hotel, Leicester, be adopted.

3. ANNUAL REPORT OF THE COUNCIL OF THE SOCIETY

Council records that 1959 has been a memorable and an outstanding year of achievement in the history of the Society. It has been a stimulating and a very busy year for all who work to further its interests and to maintain its motto of "Science is the Servant of Colour". The vast amount of voluntary service given both at home and overseas has ensured the continued expansion of the Society's interests. A welcome event in this connection was the inauguration of the West of England and South Wales Section in June 1959.

The London Congress was a most memorable occasion, being the first of its kind ever held in England. It took place in London on the 16th, 17th and 18th September 1959, when the Society was host to the International Federation of Associations of Textile Chemists and Colourists. The Congress was well attended, the lectures being held at Church House, Westminster. Enjoyable social functions took place at Guildhall and Dyers' Hall in the City of London, at the Grosvenor House Hotel, and at places of interest.

Congratulations are extended to Mr. John Boulton, 39th President of the Society, on his being the first British President of the International Federation, and on the great success of the Congress. Thanks are due to the Planning Committee and to its Chairman, Mr. Clifford Paine, and the Chairmen and Members of the Finance, Scientific, and Social Subcommittees for their valuable and successful services.

The Council is pleased to elect Past President Clifford Paine to the Honorary Membership of the Society in recognition of his valuable services to the Society and to industry. This is the highest honour Council is empowered and privileged to bestow.

The Council has awarded *two* Perkin Medals, *three* Society's Gold Medals, *four* Silver Medals, *two* Bronze Medals, and two Certificates of Merit, particulars of which are listed in the report of the Medals Committee.

It is with deep regret that the great loss is recorded which the Society has sustained by the death of Fred Smith, who was President from 1954 to 1956 and whose services to the Society were unbroken from the age of 28. Council pays warm tribute to his long and devoted services. He died on 6th October 1959.

The Prime Warden of the Worshipful Company of Dyers, Lieutenant Colonel H. F. Holme, T.D., was the principal guest at the Annual Dinner of the Society held in Leicester on 10th April 1959. Council is mindful of the consistent support so willingly accorded to our Society and its activities and records its appreciation.

The Fifteenth John Mercer Lecture was given in London on 1st May 1959, when Mr. I. S. Moll presented an excellent paper on *Aspects of Pigment Dispersion Related to Usage*. The meeting was well attended and Council acknowledges with thanks the generosity of the anonymous donor which makes possible these Mercer Lectures.

The Society took part in a Royal Society *Conversazione* at Burlington House on 9th December 1959, when an exhibition with the theme "Science in Everyday Life" was held under the auspices of the Parliamentary and Scientific Committee. The Society's colourful display showed the scope of its interest in fastness testing, and Council records its appreciation of this highly successful exhibit.

A Disperse Dyes Committee has been set up under the Chairmanship of Mr. C. L. Bird, with Mr. E. Waters as Honorary Secretary.

Donations to the Society and the continued support of the Dyers' and Finishers' Association in respect of prizes to be competed for by Junior Members of the West Riding Section are gratefully acknowledged, and Council especially thanks the firms, colleges, and technical institutions for their valuable assistance in allowing so many members of their staff to devote time to the affairs of the Society.

The twelve years service to the Society of Dr. C. J. W. Hooper are acknowledged: he has ably maintained the high standard of the *Journal* during these years, and Council wishes him well in his new venture.

In particular, Council extends thanks to authors of papers and communications, referees, and all who contribute in honorary service to the Society and to the *Journal*.

The Chairmen, Honorary Secretaries, and Committees of the Sections and the Junior Branches of the Society continue to devote a great deal of time

and energy in service to the Society. Council is greatly appreciative of their good work.

Council has pleasure in reporting the establishment of closer working arrangements with kindred bodies overseas, and in this respect is greatly assisted by the Society's International Relations Committee, who have promoted interest, understanding and good relationship with the Australian and South African Associations. Friendly co-operation continues between the American Association of Textile Chemists and Colorists and the Society.

The Society's Seventh Annual Golf Competition was held at Leek on 14th June 1959. Council thanks Mr. W. R. Leigh for arranging this successful competition and social event. The winner on this occasion was Mr. R. Jessop of Huddersfield, and he has received the miniature cup which has been presented to all winning competitors since the inauguration of the competition.

Council is pleased to report that the Society's membership continues to increase.

Membership at 1st January 1959	2931
New members elected in 1959	291
	3222
Less 148 Resignations and 14 Deaths	162
Membership at 31st December 1959	3060
Journal Subscribers at	
31st December 1959	1429
Total	4489

Reports on Activities of Committees

FINANCE AND GENERAL PURPOSES COMMITTEE—This Committee has been very active during the year in arranging for a Memorandum of Agreement between the American Association of Textile Chemists and Colorists and the Society which has facilitated an interim settlement between the two societies in regard to the production of the *Colour Index*.

Further consideration has been given to the method of financing the *Colour Index* Supplement. In addition, the Committee has given very careful consideration to ways and means of producing more revenue, and of effecting economies in the affairs of the Society. A system of budgetary control has also been reviewed, and it is pointed out that, although a deficit appears in the working of the Society, the liquid position remains very satisfactory.

SOCIETY'S MEDALS COMMITTEE—The following recommendations of the Committee were approved by Council—

<i>Perkin Medals</i>	Cecil John Turrel Cronshaw John Thompson Marsh
<i>Gold Medals</i>	Ian Durham Rattee William Elliot Stephen Heinrich Ris

Silver Medals

Geoffrey Gordon Bradshaw
Herbert Frederick à Brassard
Norman Hamer
John William Reidy
William Robert Leigh
John Rayment

Bronze Medals

Certificates of Merit

The late Abraham Burawoy
S. J. Pentecost

Details of the citations have been published in the January 1960 *Journal*. The awards will be presented at the Society's Annual Dinner at Leeds on 29th April 1960.

WORSHIPFUL COMPANY OF DYERS RESEARCH MEDAL COMMITTEE—In accordance with the decision of the Society's Adjudication Committee, based on a survey of eligible papers in the *Journal* within the prescribed period, Council has recommended that, as no paper appeared to be of sufficient merit, the Research Medal be not awarded for this year. The recommendation has been accepted by the Court of the Worshipful Company of Dyers.

DIPLOMAS COMMITTEE—Five recommendations were approved by Council for conferment of Fellowships. As a result of the examinations seventeen recommendations for the award of the Associateship were accepted by Council.

PUBLICATIONS COMMITTEE—The Committee has a monthly meeting to consider papers submitted for publication, to consider referees' reports, to appoint book reviewers, and to pass the *Journal* galley proofs.

During the first half of the year a detailed investigation of the cost and possible developments of the *Journal* was undertaken.

The printers' strike caused delays in the publication of the August and subsequent issues of the *Journal*, but the Committee is pleased to report that normal publication dates are almost achieved.

The Committee places on record its appreciation of the action of Mr. C. L. Bird and Mr. C. O. Clark in assuming responsibility for editing the Papers and Communications and the Abstracts respectively on the resignation of the Editor in October 1959.

PRESERVATION OF HISTORICAL RECORDS COMMITTEE—The catalogue of the Society's collection of historical records is almost complete and consideration is being given to proposals for arranging a suitable display.

Colour Index EDITORIAL BOARD—At a meeting with Mr. W. D. Appel and Mr. G. Paine of the A.A.T.C.C., future policy and working relations between the two editorial bodies were discussed and complete agreement was reached on all outstanding editorial problems. The Board reports that material for the Supplement is coming in steadily.

FASTNESS TESTS CO-ORDINATING COMMITTEE—Mr. K. McLaren succeeded Dr. P. W. Cunliffe as Chairman and Miss Esmée Smith was appointed Honorary Secretary.

An important event was the production of the Second Edition of *Standard Methods for the Determination of the Colour Fastness of Textiles*, which will be published in 1960. Incorporated are all the alterations and additions which have been made since the First Edition was published in June 1955.

The Fastness Tests Co-ordinating Committee has submitted a proposal to B.S.I. that I.S.O. should create a Technical Committee for Colour Fastness.

A new subcommittee has been appointed to study two tests concerned with dry cleaning.

The Pleating Fastness Subcommittee, now renamed the Heat Treatments Subcommittee, has devised a test for dry-heat setting using molten metal as the heat-transfer medium. It is hoped that this test will be accepted by I.S.O.

The Burnt-gas Fumes Subcommittee has devised a new test in which the destructive agency—nitrogen oxides—is generated from a mixture of sodium nitrite and phosphoric acid instead of by burning coal-gas as in the 1955 method.

The Alkaline Milling Fastness Subcommittee has been formally disbanded, but the Light, Perspiration, and Washing Subcommittees are active.

Through Council the F.T.C.C. expresses its thanks to all Subcommittees.

Publications during the year included a translation into French of a review of light fastness testing, and into Italian of an article on weathering fastness testing.

TERMS AND DEFINITIONS COMMITTEE—The Committee has proposed about thirty terms, and close collaboration between the Committee and the Textile Institute has been maintained. The Committee invites suggestions of terms requiring definition. Mr. S. R. Cockett resigned as Chairman and his place has been taken by Mr. J. S. Ward.

REVIEW OF TEXTILE PROGRESS—Mr. G. S. J. White, who has been Chairman for the past ten years, has resigned now that the ten volumes of the *Review* have been completed. Council thanks Mr. White for his long service as Chairman of the Committee and is pleased to note that he is continuing as an ordinary member.

MOH AND DERMSTID BEETLE PROOFING—The immediate work of this Committee, a joint committee with the Textile Institute, is now covered by the work of the International Wool Textile Organisation. Accordingly the Committee has been disbanded, and Council thanks members of the Committee.

DATA ON IDENTIFICATION OF DYES ON TEXTILE FIBRES—The Committee has given consideration to amendments and additions to the established schemes for the identification of dyes on wool and cellulosic fibres, as well as to the possibility of drawing up new schemes for identification of dyes on the newer synthetic fibres and on the cellulose acetates, but a great deal of experimental work is still required.

INTERNATIONAL RELATIONS—The Committee is planning personal liaison with individuals and with organisations similar to our own Society in other countries, in order to strengthen the bonds and promote closer working arrangements with the Society.

Activities of Sections

Council recognises with admiration the strength of the support given by Sections, and in thanking all concerned for their interest and enthusiasm, mention is made of the following activities, which are in addition to the much-appreciated lectures and social events arranged during the year.

HUDDERSFIELD—Mr. G. H. Binns, Chairman of the Section, reports that eight well-supported lectures have been given and that particularly successful events were the visits exchanged with the Huddersfield and Halifax Textile Societies and the Huddersfield Optical Society. Another successful occasion was the showing of a series of colour films following the Section's Annual General Meeting. The locally incurred expenditure of the Section for the year was £47 17s 8d, and there are now 168 members on the register.

LONDON—Mr. R. Woods, the Section Chairman, records thanks to the Royal Society for the continued privilege of the use of their Rooms at Burlington House for the Section's lectures. Joint meetings in London with the Textile Institute, the Guild of Dyers and Cleaners, and the Clothing Institute have contributed to the advancement of mutual interests. Among the activities of the Section is the Luton Lecture, and the interest of Vice-president R. C. Oakley and his support is gratefully acknowledged. The Society's Mercer Lecture given in London and the Seventh London Lecture on *Colour and Design in Wallpapers* were successful events. The locally incurred expenditure of the Section for the year was £83 8s 3d, and there are now 227 members on the register.

MANCHESTER—The Section Chairman, Mr. H. A. Turner, reports the success of the Symposium on *Reactive Dyes*, and a number of lectures by distinguished speakers have attracted good audiences. In particular has the Manchester Junior Branch excelled itself by substantially increasing its membership, and the papers presented throughout the year have been of high quality. The locally incurred expenditure of the Section (including the Junior Branch) for the year was £100 6s 6d, and there are now 710 members on the register.

MIDLANDS—Mr. A. S. Cluley, the Section Chairman, reports that his Section was host to the Society on the occasion of the Society's Annual General Meeting and Dinner held in Leicester in 1959, and another important occasion was the Half-day Symposium on *Acrylics in Knitwear*, which was well attended. Successful lectures and functions have contributed to the well-being of the Section. The locally incurred expenditure of the Section for the year was £58 0s 2d, and there are now 328 members on the register.

NORTHERN IRELAND—It is with regret that we report the death of Mr. W. Graham on 27th February 1960. The late Mr. W. Graham, as Chairman of the Section, had reported that lectures, sporting and social activities had been very successful. The lecture on *Theory and Practice of Sodium Chlorite Bleaching* attracted an audience of more than 100.

The Belfast College of Technology had expressed high appreciation of the annual gift of five guineas to be awarded as a prize to a student taking the Bleaching, Dyeing, and Finishing course. The locally incurred expenditure of the Section for the year was £37 16s 0d, and there are now 149 members on the register.

SCOTTISH—Mr. J. G. B. McCallum, Chairman of the Section, thanks the Governors of Paisley Technical College, the Royal Technical and Scientific College, Glasgow, and the Scottish Woollen Technical College, Galashiels, for their assistance with accommodation and other facilities for meetings. An award of a Junior Essay Prize has been much appreciated. The Section thanks the lecturers who contributed to a successful session. The locally incurred expenditure of the Section (including the Junior Branch) for the year was £68 13s 9d, and there are now 250 members on the register.

WEST OF ENGLAND AND SOUTH WALES—The inaugural meeting with an attendance of about 100 was held on 26th June 1959 at Bristol, when Dr. G. Welford was appointed Chairman of the Section. The first lecture was given by Dr. T. Vickerstaff on 29th October, the title of the lecture being *Application of Research in the Colouring Industries*. As an experiment, meetings are being held in different towns in the area, including South Wales. There was no locally incurred expenditure of the Section, and there are now 66 members on the register.

WEST RIDING—Mr. J. Rankin, Chairman of the Section, has drawn attention to two events which were outstanding, in that each attracted an attendance of 150. These were the One-day Conference on *Crease Retention and Crease Shedding* and the Geigy Bicentenary Lecture, when the subject was *The Kinetics of Wool Dyeing with 2:1 Metal Complexes*, the lecturer being Dr. H. Ackermann. The lectures have covered a wide variety of subjects and the social sphere has been an outstanding success. The locally incurred expenditure of the Section (including the Junior Branches) for the year was £69 11s 0d, and there are now 517 members on the register.

Representation on External Bodies

Council very much appreciates the work of the Society's representatives on the following External Bodies, who continue to be active, and a report of their work will be published in the *Journal*.

Bradford Institute of Technology—Governing Body

British Biological Stains Commission

British Standards Institution—

Colouring Matters for Use in Foodstuffs

Cotton Industry Committee

Flammability of Fabrics

Letterpress Inks

Linen Industry Committee

Methods of Test for Surface Active Agents

Proofed Clothing—Dyeing and Finishing Processes Committee

Pyridine

Rayon Industry Committee

Rotproofing of Textiles other than Clothing

Silk Industry Committee

Steering Committee T/-/8

Subcommittee and Panel on Standards for Anodic Oxidation Finishes for Aluminium and Aluminium Alloys

Textile Divisional Council

Textile Machinery Industry Committee

Wash Fastness of Buttons

Wool Industry Committee

Chemical Society Joint Library Committee

City and Guilds of London Institute—

Dyeing Advisory Committee

Consultative Committee for Textile Subjects

Faraday Memorial Committee

Manchester College of Science and Technology—Court of Governors

National Illumination Committee of Great Britain and the Subcommittee on Colorimetry and Colour Rendering

Parliamentary and Scientific Committee

Regional Advisory Council for Technical and Other Forms of Further Education for Manchester and District—

Post Advanced Textiles Advisory Committee

Post Advanced Chemistry Advisory Committee

Royal Society—National Committee for Chemistry

Royal Technical College, Salford—Applied Sciences Advisory Committee

Society of Leather Trades Chemists—Committee for Fastness Properties of Dyes on Leather

Textile Institute—

Technical Committee "D"

Textile Terms and Definitions Committee

Unification of Testing Methods Committee

Yorkshire Council for Further Education—

County Advisory Committee for Chemistry

County Advisory Committee for Textiles

* * *

The Honorary Secretary, Mr. L. MORTON WOOD, said that he had pleasure in presenting once again the Annual Report of the Council of the Society, which had been circulated to all members. He made no excuse for drawing attention once more to the tremendous amount of voluntary work undertaken, and felt that therein lay the Society's strength. He thanked the voluntary workers, and the firms who had made it possible for them to undertake that work.

Referring to the resignation of the Editor of the *Journal*, Dr. C. J. W. Hooper, in October last, Mr. Wood said that a new Editor—Dr. M. Tordoff—had now been appointed, and he would commence his duties next August. He felt sure that Dr. Tordoff would be happy in the Society and would make a success of his undertaking. He thanked the Acting Editors, Mr. C. L. Bird and

Mr. C. O. Clark, for stepping into the breach at short notice. Together with the Publications Committee, led by Mr. A. Thomson, they had made a magnificent effort in keeping the *Journal* up to standard—indeed, he had heard it said that it was even better than before.

Mr. Wood welcomed the new West of England and South Wales Section, which had been formed during the year, and wished it every success. It would, no doubt, have certain "teething" troubles, but it would be able to count on the help and guidance of Sections long-established.

He also announced a new committee—the Disperse Dyes Committee—which had got off to a good start. It was apparent, he felt, that the status and standing of the Society was increasing. This had been evident at the London Congress in September last.

In conclusion, he wished to thank the Society's General Secretary, Mr. John Nicholls, for his industrious work during the past year at considerable detriment to health and leisure; and he also thanked the staff at Dean House. He had great pleasure in formally proposing the adoption of the Annual Report of Council.

Mr. C. L. BIRD seconded the adoption, which was carried *nem. con.*

4. PRESENTATION OF BALANCE SHEET, STATEMENT OF ACCOUNTS, AND AUDITORS' REPORT FOR THE YEAR ENDED 31ST DECEMBER 1959

The Honorary Treasurer, Mr. A. WADDINGTON, said that, as in previous years, he did not propose to go through each individual item. The deficit on the year's working followed the pattern of the last three years, and those present at the last Annual General Meeting would remember that he had then issued a warning that the gap of surplus income was closing. It had since closed and there was now a deficit.

He wished to mention two items on which there was information which was not apparent in the Accounts. In the General Revenue Account, under the heading "Income", was "Accrued Interest on Realisation of National Savings Certificates". This was a non-recurring item. The period for which the Savings Certificates had been invested had now expired, notice of withdrawal had been given, and the money would shortly be reinvested at a higher rate of interest.

The question of the future finances of the Society was concerned with Council's recommendation for increasing the annual subscriptions, which was on the agenda and was to be considered later.

With further reference to the Balance Sheet, it would be found under the heading of "Surplus: Accumulated Fund" that, after deduction of the year's deficit, there was an addition to the Fund in respect of the *Colour Index* Account. This addition consisted of expenses not chargeable against the joint account with the American Association of Textile Chemists and Colorists, owing to an arrangement made in about 1952, but as from 1st January 1959 this old agreement with the American Association had been substantially modified. The Society had now taken over a

number of the Association's volumes of the *Index* and, apart from the fact that the Society had agreed to a subscribers' minimum rate, it could now dispose of its volumes as it wished, any resultant surplus being the property of the Society.

The Balance Sheet included an item of £1,638 for the I.F.A.T.C.C. London Congress. This sum had now been refunded to the Society: the firms who had kindly guaranteed the costs of the Congress had made their contributions and the Society had been reimbursed. "Additions and Amendments" was a new item. The Society was now publishing *Additions and Amendments* and eventually would publish a supplement to the *Colour Index*. Assistance, for which the Society had agreed an annual payment, would be given by the A.A.T.C.C. The information would then be dealt with by the *Colour Index* Editorial Board, and a supplement would eventually be issued.

Last year's comparison figures were published in the Accounts, and Mr. Waddington formally moved the adoption of the Accounts and Balance Sheet.

Mr. A. THOMSON said that he had great pleasure in formally seconding the adoption.

In reply to a question, the Honorary Treasurer stated that the *Colour Index* volumes in storage were insured against all possible hazards.

Mr. H. FOSTER quoted expenditure for the year 1955 as compared with 1959, and also expressed the wish that consideration be given to the question of bringing investments into the Balance Sheet at their current value.

Mr. J. W. REIDY drew attention to the depreciation of office equipment.

The Honorary Treasurer, in reply, explained that there had been additions to the office equipment during the year. These were necessary, but it was hoped that the need for additions would gradually disappear. The equipment was being written off at 10% per annum.

Mr. H. FOSTER said that expenditure attributed to the various Sections in the Annual Report of Council amounted to a sum in excess of Sectional costs shown in the Balance Sheet.

The Honorary Treasurer explained that the Sectional expenses referred to in the Annual Report were those incurred by the Sections and reimbursed by him. The total in the Balance Sheet included both the amounts reimbursed and the accounts paid by Head Office for work done on behalf of the Sections. The latter covered such items as printing, stationery, circulars, and postage.

Dr. P. W. CUNLIFFE asked for more details of the Fastness Tests Account.

The Honorary Treasurer said that, at the beginning of the year, the stock was £1,200 worth of material that had not been cut up into bunches. The stock was valued at cost.

The Annual Accounts were formally adopted.

5. RECOMMENDATION OF THE COUNCIL OF THE SOCIETY TO INCREASE THE MEMBERSHIP SUBSCRIPTIONS

The Honorary Treasurer, Mr. A. WADDINGTON, in proposing the increase in the annual subscriptions, said that he wished to assure members that the

Balance Sheet as at 31 December 1959

[illegible]

AUDITORS' REPORT

We have examined the above Balance Sheet with the Books and Documents of the Society produced to us. In our opinion the above Balance Sheet is properly drawn up so as to exhibit a true and fair view of the state of the Society's affairs according to the best of our information and the explanations given to us and as shown by the Books of the Society.

RAWLINSON, GREAVES & MITCHELL
Chartered Accountants
Auditors

Bradford
3 February 1960

Consolidated General Revenue Account

for the Year ended 31 December 1959

31.12.58	EXPENDITURE	£	31.12.58	INCOME	
	Salaries, Wages, National Insurance and Honoraria	9,568	8,173	Membership Subscriptions Journal	
10,116	Rent, Rates and Insurance	799		Income from Journals, Reprints, Combined Reports, Advertisements, etc., as adjusted for Opening and Closing Stocks	
835	Electricity and Heating	182	17,351	Investment Income (Gross)	
181	Telephone	584	703	Accrued Interest on Realisation of National Savings Certificates	
358	Printing and Stationery	1,249		Interest on Bank Deposit Account	
1,575	Postages	612	7	Balance on account "Fastness Tests"	
576	Hire of Rooms for Meetings, etc.	388	1,800	Donations	
326	General Travelling and Hotel Charges (Including £153 relating to 1958)	883	587	Net Deficit for the Year	
273	Incidental Office Costs	206			
269	Depreciation of Office Furniture and Equipment	350			
354	Bank Charges	61			
76	Audit	250			
320	Repairs and Renewals	92			
116	Medals and Diploma Costs	233			
273	Sectional Costs	1,527			
1,547	Printing and Publishing Journal and other Publications	13,860			
15,577	Abstractors' Fees and Book Reviews	596			
679	Printing and Publishing Colour Index	—			
38	Colour Index—Administration, Advertising, Shipment and Despatch Additions and Amendments	—			
3,841	Expenses incurred for Colour Index but not allowable per Agreement with A.A.T.C.C.	—			
47	Colour Index Celebration Dinner	—			
293	Annual Meeting and Dinner	154			
73	Summer School	125			
155	International Federation Subscription	—			
116	Peter Griess Symposium	—			
49		—			
38,068		31,719			
	Less—Expenditure incurred above and allocated to Colour Index (£1,295) and Additions and Amendments (£1,065)	2,360			
9,297					
£28,771		£29,359	£28,771		

COLOUR INDEX (2nd EDITION) ACCOUNT

as at 31 December 1959

	£		
Balance 1st January 1959	6,004	Income	
Expenditure	1,732	Stock of Colour Index at 31st December 1959	
Amounts Due to A.A.T.C.C. under the modified agreement	402	(At Value agreed with A.A.T.C.C.)	
Expenses, incurred since 1946 for Colour Index and not allowable per agreement with A.A.T.C.C., now charged against Colour Index Income	1,527		
Additions and Amendments			
Amounts received previously for Additions and Amendments now transferred to that Account	260		
Balance, transferred to Development Fund	1,372		
	£11,297		

GEORGE DOUGLAS ACCUMULATION FUND ACCOUNT

31.12.58		£	31.12.58		£
105	Lecture Expenses	6		Balance per Accumulated Income Account	231
230	Balance per Accumulated Income Account	341	276	1.1.59	56
				Lecture Expenses Refunded	
			59	Investment Income (Gross)	60
				3% British Transport Stock 1978/88	
<u>£335</u>		<u>£347</u>	<u>£335</u>		<u>£347</u>

GEORGE DOUGLAS LECTURE FUND

	Balance 1958	Alterations	Balance 1959	Middle Market Value 1958	Middle Market Value 1959	Middle Market Quotation
3% British Transport Stock 1978/88	<u>£2,000</u>		<u>£2,000</u>	<u>£1,314</u>	<u>£1,334</u>	67½

KNECHT MEMORIAL FUND

3% Southern Rhodesia 1971/73	<u>£100</u>		<u>£100</u>	<u>£73</u>	<u>£71</u>	72
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SCHEDULE OF FOUNDATION FUND ACCOUNT INVESTMENTS

	£	£	£	£
National Savings Certificates				
10a. Units 8th Issue	500	—	500	—
Proceeds of Realisation not yet re-invested	—	500	—	500
3% Savings Bonds 1960/70	70	70	55	82½
Treasury 3½% Stock 1979/81	36	36	29	78½
3% British Electricity Guaranteed Stock 1968/73	1,245	1,245	1,344	1,376
	<u>£1,851</u>	<u>£1,851</u>	<u>£1,928</u>	<u>£1,962</u>

SCHEDULE OF ACCUMULATED FUND INVESTMENTS

	£	£	£	£
3% British Transport Guaranteed Stock 1978/88	2,599	2,599	1,831	1,858
2½% Funding Stock 1956/61	750	750	721	739
3½% Conversion Loan 1961 or after	557	557	362	357
3% Savings Bonds 1960/70	2,750	2,750	2,224	2,277
3% Funding Stock 1966/68	750	750	624	636
National Savings Certificates				
15a. Units 7th Issue	375	—	375	—
3% British Gas Stock 1990/95	2,000	2,000	1,328	1,344
3% Treasury Stock 1966 or after	1,957	1,957	1,168	1,145
3% Southern Rhodesian 1971/73	911	910	661	649
York County Savings Bank	4,581	4,718	4,581	4,718
		Interest credited during the year		
		£129		
		Defence Bonds Interest credited		
		during the year £8		
3% British Electricity Guaranteed Stock 1968/73	1,496	1,496	1,159	1,187
Post Office Savings Bank	1,158	1,187	1,158	1,187
		Interest credited during the year		
		£29		
Chesterfield Corporation 3% Stock 1960	994	994	970	990
Treasury 3½% Stock 1979/81	1,007	1,007	802	819
3½% Defence Bonds	220	220	220	220
	<u>£22,105</u>	<u>£21,895</u>	<u>£18,184</u>	<u>£18,126</u>

decision to recommend the increases had not been arrived at without very careful consideration, particularly in the Finance and General Purposes Committee and subsequently in Council.

The affairs of the Society fell into two main groups, viz. (1) the *Journal*, and (2) general administration. As far as the *Journal* was concerned, it had been decided without dissent that it must be maintained at its present high standard and continue to be recognised as the leading publication of its kind. In the case of the second group, the work undertaken by the Society was increasing and the working expenses would be in excess of the proposed increased subscriptions. The cost of the *Journal* depended to a large extent on the amount of text published, and it was anticipated that, in 1960, more would be published than in 1959, with consequently increased cost.

In 1959 accumulated income from Savings Certificates was £300, but this was a non-recurring item. The surplus on the Fastness Tests Account fluctuated.

Mr. Waddington pointed out that Council had not taken the easy course of saying: "We must have another £4,000 to cover the deficiency in income, so we will increase the subscriptions accordingly." Adoption of the resolution should bring in, in a full year, a further £2,800, and Council and the Finance and General Purposes Committee had decided that they must make their contribution to the extent of £1,200, by finding other forms of revenue or reducing expenditure.

The Honorary Treasurer reminded members that 90% of them received income-tax rebate on subscriptions, and that, in fact, four guineas less tax was less than the three guineas which they had paid before income-tax relief had been granted in the previous year. He hoped that this might be some consolation.

Summarising the matter, he said that, if the Society was to maintain its present efficiency of working and the high standard of the *Journal*, income would have to be raised, and he could not see any possible way other than by increasing subscriptions, but no stone would be left unturned in order to increase further the income of the Society from every other available source. Mr. Waddington then proposed that, as from 1st January 1961, the membership subscriptions be as follows—Ordinary Members £4 4s 0d; Members between 21 and 25 years of age £2 12s 6d; and Junior Members £1 1s 0d.

Mr. R. J. HANNAY, in seconding the proposal, said that, over the last two years, he had consistently opposed any resolution that membership subscriptions should be increased. He had had the honour of serving continuously on the Finance and General Purposes Committee and on Council for longer than most members present; he had been, in one capacity or another, on the Council for the last ten years and, up to the present year, he had not considered that Council had any case for recommending a higher subscription. The fact that costs were going up had not, in his view, justified an increase if some economies in running costs could be achieved.

It was a good thing that there were members who were prepared to stand up at the Annual General Meeting and criticise the Society's expenditure, because they kept officers and committees on the alert.

Probably the biggest cause of loss of income was to be found in the difference between receipts from and expenditure on the *Journal*. For many years the *Journal* had contributed about £2,000 per annum to the running of the Society. Owing to increased printing costs, this contribution had now completely disappeared and money must be found to replace it.

It was evident that, if the Society was to maintain its position and prestige in the scientific world, additional finance was necessary. Members were being asked to contribute by increased subscription about 50% of the estimated deficiency, leaving Council to find by some means the other 50%.

It was hoped that, in the years to come, the *Colour Index* would bring in an appreciable amount of money, but it would be wrong to use that income for current expenditure: it was for development, and would be put on one side for that purpose. He was confident that the proposal was in the best interests of the Society and that was why he was seconding the motion.

Mr. D. HAIGH, in opposing the motion, said that there was little in the Annual Report of Council or the Balance Sheet to support what the Honorary Treasurer and Mr. Hannay had said. He would like Council to put a reasoned case before every member of the Society before a decision was made. Very little, if any, effort had been made to discuss the motion in the Sections before the Annual General Meeting. The increased subscriptions might result in a considerable fall in membership. Even last year, without an increase in subscription, 148 members had resigned.

The President, intervening, pointed out that these resignations were due to changes in occupation.

Mr. Haigh, continuing, said that he would support every measure to maintain the *Journal* at a high level, but did many of the members really require the Abstracts? Was it possible to make a separate charge for Abstracts? It was strange that investments of £25,000 were only worth £20,000 at the present day. Surely there were ways of achieving a better return? He moved an amendment to refer the motion back to Council for further consideration.

Mr. H. FOSTER seconded the amendment.

Mr. C. P. ATKINSON asked what was to happen to the option of 40-year and 50-year members with reference to subscriptions.

The President replied that this would remain unchanged.

Dr. H. W. ELLIS said that the question of increasing membership subscriptions must have been given deep consideration by those concerned. Every member had been notified of the fact that it was to be raised at the Annual General Meeting, and it was for the individual member, if he felt

strongly about it, to attend, express his views and vote, as he thought fit.

Mr. J. T. MARSH said that he subscribed to six or seven different societies, not all of them scientific, but for many years the Society of Dyers and Colourists had been his intellectual and spiritual home. No other society with which he was associated gave him anything like the same value for money. Many organisations had increased their subscription rates without complaint. If the *Journal* was to be extended in scope, the proposal to increase subscriptions was a very wise one.

Mr. J. W. REIDY said that a start might be made, in obtaining additional revenue, by raising the charge for advertisements. As there had been criticism of lavish expenditure, it had been decided to give the Sectional expenditure in the Annual Report of Council, and it was evident that the Sections were attempting to keep their costs down.

Mr. R. A. PEEL said that there were many younger members of the Society for whom the cost of living was very high and who could not claim the full income-tax deduction; they were going to find it very hard to pay an extra amount. Up to the age of thirty a member should pay not more than three guineas.

The President, in closing the discussion, said that, during his two years of Presidency, he had become convinced that the work done by the Society—and, in consequence, the standing which it and its *Journal* enjoyed all over the world—represented something for which they would have to pay a great deal more in many other organisations. The *Journal* was doing a job *par excellence*. It was true that young members would have to pay a guinea more, and costs were going up, but so were salaries in proportion. The Society was the only one of its kind that had not put up its subscription very sharply since the war. Members were getting more than value for their money. While he had been President, he had had the opportunity of attending meetings of the Finance and General Purposes Committee, and of taking part in initiating the budgetary-control system. Any savings that could be effected were limited in extent. It was no use hoping that Council could reconsider the matter and produce a budget which would enable the Society to run at a much lower financial cost than at the present time.

The amendment was put to the meeting and was declared lost.

The proposition was then put to the meeting and carried, it being—

Resolved that, as from 1st January 1961, the membership subscriptions be as follows—

	£	s	d
Ordinary Members	4	4	0
Members between			
21 and 25 years of age	2	12	6
Junior Members	1	1	0

6. ELECTION OF PRESIDENT FOR THE FORTHCOMING YEAR

The President said that it was his very pleasant duty to propose that the new President of the Society should be Mr. F. Malcolm Stevenson.

A year ago he had proposed Mr. Stevenson as President-elect, and he would not repeat what he had said then. It sufficed to say that he had had more opportunities of appreciating Mr. Stevenson's generosity, technological outlook and long-standing interest in the Society. As President-elect, Mr. Stevenson had never missed an opportunity of helping the Council. In proposing Mr. Stevenson's nomination, he would like to thank him very much for the personal help given to himself as President during the last twelve months.

Mr. W. PENN, Vice-president of the Society, said that he wished to support all that Mr. Boulton had said. The Midlands Section were delighted that a man of Malcolm Stevenson's calibre was to be President of the Society. They were sure that he would perform his duties in a way which would carry him through a wonderful year of office.

The proposition, on being put to the meeting, was carried with acclamation.

Mr. Stevenson was invested with the chain of office and presided over the subsequent proceedings.

The President, Mr. F. M. STEVENSON, in thanking the members for the high honour they had conferred upon him, said that he would have to lean very heavily on Mr. Boulton. His first, and pleasurable, duty was to invest Mr. Boulton with the Past-president's Badge. Mr. Boulton had been President for two years, and that was a considerable burden on any man. He had carried the Society's flag to the farthestmost points of the earth and had enhanced considerably the standing of the Society; they should offer him their very best thanks.

The retiring President, Mr. JOHN BOULTON, said he would like to say how much his two years of office had meant to him in interest, exhilaration, and inspiration. A great deal had been done during that time, but it was not the work of the President, but of a team of people, whom he thanked and who, he was sure, would give their new President the same support.

7. ELECTION OF PRESIDENT-ELECT

The President said that he presented for the members' acceptance the name of Mr. R. J. Hannay as President-elect in 1961. They had heard Mr. Hannay say that he had been continuously on the Council, on the Finance and General Purposes, and on other committees for the past ten years: this was an exceedingly fine "school" from which to elect a President of the Society, and he had the utmost confidence in nominating Mr. Hannay.

Mr. J. BARRITT said that he was most grateful for having been given the opportunity to second the nomination. He had known Mr. Hannay for a very long time. Mr. Hannay was a man of great ability and integrity who would work very hard and do all the Society could wish.

The nomination was approved.

The President-elect, Mr. R. J. HANNAY, said he thanked the members very much indeed. His one regret was that his father had passed away before the honour had been accorded.

8. ELECTION OF ONE VICE-PRESIDENT

The General Secretary gave the result of the ballot for one Vice-president, which was as follows—

Mr. G. S. J. White	549 votes
Mr. C. O. Clark	479 votes

The President said that he had much pleasure in declaring Mr. White elected a Vice-president.

9. ELECTION OF FOUR ORDINARY MEMBERS OF COUNCIL

The General Secretary reported that 1046 ballot papers had been received, of which three were spoilt. The numbers of votes recorded for each candidate were as follows—

Dr. C. B. Stevens	607 votes
Prof. R. H. Peters	595 votes
Dr. C. H. Giles	542 votes
Mr. R. K. Fourness	492 votes
Mr. H. R. Hadfield	457 votes
Mr. S. R. Meadows	293 votes
Mr. F. V. Davis	253 votes
Mr. R. Hawthorn	218 votes
Mr. E. E. Bowker	208 votes
Dr. W. L. Lead	192 votes

The counting of the votes, by two members of the staff of the Auditors to the Society, had been supervised by Mr. R. Grice.

The President declared the following elections to Council—

Mr. R. K. Fourness
Dr. C. H. Giles
Prof. R. H. Peters
Dr. C. B. Stevens

and welcomed the new members.

10. VOTE OF THANKS TO SCRUTINEER

A vote of thanks to the supervising scrutineer, Mr. R. Grice, was proposed by Mr. A. THOMSON, seconded by Mr. J. PORTER, and carried.

11. VOTE OF THANKS TO RETIRING VICE-PRESIDENT AND ORDINARY MEMBERS OF COUNCIL

Mr. R. C. OAKLEY proposed a vote of thanks to the retiring Vice-president, Mr. H. A. Turner, and to the retiring Ordinary Members of Council—Dr. G. T. Douglas, Mr. F. Farrington, Mr. N. Hamer

and Dr. L. Peters, who, he said, had served the Society very well. This was seconded by Mr. A. S. CLULEY and carried.

12. ELECTION OF HONORARY TREASURER

The President said that it gave him great pleasure to present the unanimous recommendation of Council, that Mr. A. Waddington be asked to continue to serve the Society as Honorary Treasurer.

Mr. Waddington was declared unanimously re-elected.

13. ELECTION OF HONORARY SECRETARY

The President said that he had also great pleasure in presenting Council's nomination of Mr. L. Morton Wood for re-election as Honorary Secretary.

Mr. L. Morton Wood was unanimously re-elected Honorary Secretary.

14. APPOINTMENT OF AUDITORS

Mr. J. G. HOPKINSON proposed that Messrs. Rawlinson, Greaves & Mitchell, Chartered Accountants, be re-appointed Auditors to the Society for the year 1960. This was seconded by Mr. G. H. BINNS and carried.

15. VOTE OF THANKS TO THE CHAIRMEN

Mr. G. S. J. WHITE said that it gave him pleasure to move a vote of thanks to the chairmen of the meeting, Mr. J. Boulton and Mr. F. M. Stevenson. The Annual General Meeting had been an exciting and interesting occasion. The retiring President, who had taken them through all the pomp and ceremony of the London International Congress last September, had been, during the last few minutes of his Presidency of the Society, the recipient of thunderbolts from both sides of the House; but Mr. Boulton—as was his custom—had dealt with it suavely, and one saw signs of this quality in their new President, Mr. Malcolm Stevenson. Mr. White thanked both these gentlemen for the excellent manner in which they had conducted the Society's affairs that afternoon.

The vote of thanks was carried with acclamation. The meeting then terminated.

Annual Dinner

The Seventy-sixth Annual Dinner of the Society was held at the Queen's Hotel, Leeds, on Friday evening, 29th April 1960, under the presidency of Mr. F. M. Stevenson.

The principal guest was Sir Donald Kaberry, Bt., T.D., M.P. Among other guests were The Right Honourable Lord Milner of Leeds, Mr. R. F. Rothwell (*Prime Warden of the Worshipful Company of Dyers*), Mr. John Boulton (*President of the International Federation of Associations of Textile Chemists and Colourists and Immediate Past President of the Society*), Dr. E. G. Edwards (*Principal of the Bradford Institute of Technology*), Mr. P. J. Gay (*President of the Oil and Colour Chemists' Association*), Mr. L. M. Wood (*President of the Dyers and Finishers Association and Honorary*

Secretary of the Society), Mr. J. J. Priestley (*Representing the President of the Society of Chemical Industry*), Sir William Garrett (*Chairman of the Association of British Chemical Manufacturers*), Mr. G. D. Wade (*Vice-president of the Dyers and Finishers Association*), Mr. B. Turner (*Immediate Past President of the Bradford Textile Society*), Mr. H. M. Blyth (*Secretary of the British Colour Council*), Mr. G. A. Spencer (*Chairman of the British Cotton and Wool Dyers' Association*), Dr. A. B. D. Cassie (*Director of Research, Wool Industries Research Association*), Mr. J. R. Ruck-Keene (*General Secretary of the Chemical Society*), Mr. R. H. Hamblin (*General Secretary of the Oil and Colour Chemists' Association*), Dr. K. W. Luckhurst (*Secretary of the Royal Society of Arts*),

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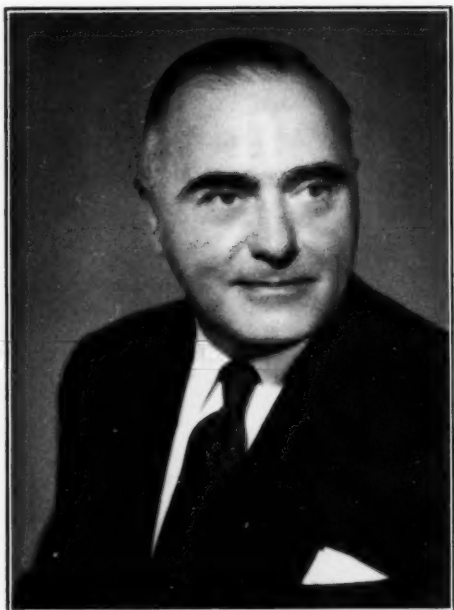
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CLIFFORD PAINE
B.Sc., F.R.I.C., F.S.D.C.

Honorary Membership in high appreciation of his
devoted service to the Society



CECIL JOHN TURRELL CRONSHAW
D.Sc.(Hon.), B.Sc., F.R.I.C., M.I.Chem.E., F.T.I.,
F.R.S.E., A.M.C.T.(Hon.), F.S.D.C.

Perkin Medal as a leader under whose enthusiastic
guidance the phthalocyanine pigments and derived
textile dyes were first made available and their
basic constitution established



JOHN THOMPSON MARSH
M.Sc., F.R.I.C., F.T.I., F.S.D.C.

Perkin Medal for outstanding contributions to the
practice and the literature of textile chemistry



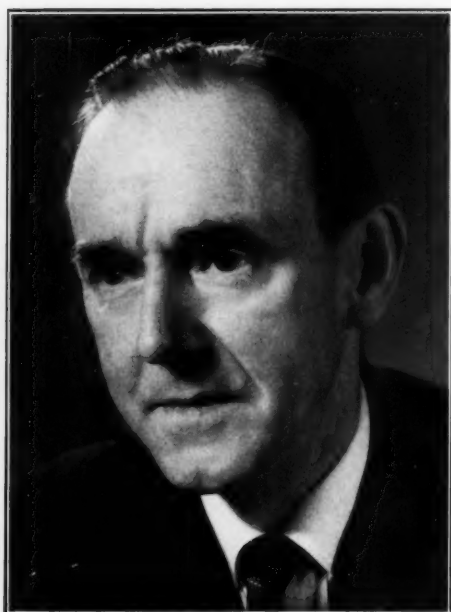
IAN DURHAM RATTEE
B.Sc., A.R.C.S.

Gold Medal for the invention and development of
the first practical system of colouring cellulosic
fibres with reactive dyes



HEINRICH RIS
Dr.Phil.

Gold Medal for distinguished contribution made
over many years to international relations amongst
dyers and colourists



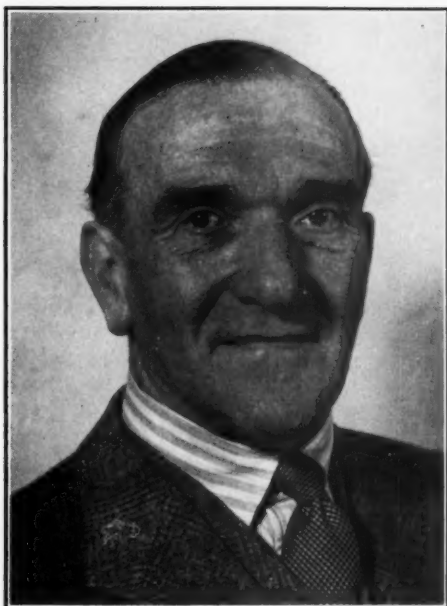
WILLIAM ELLIOT STEPHEN
B.Sc., Ph.D.

Gold Medal for the invention and development of
the first practical system of colouring cellulosic
fibres with reactive dyes



GEOFFREY GORDON BRADSHAW
 M.Sc., A.R.I.C., F.S.D.C.

Silver Medal for valuable services to the Society and particularly as a member of the Fastness Tests Co-ordinating Committee and the Publications Committee



HERBERT FREDERICK A BRASSARD
 F.S.D.C.

Silver Medal for valuable services to the Society and for the stimulation he has afforded the Society in many aspects of its work



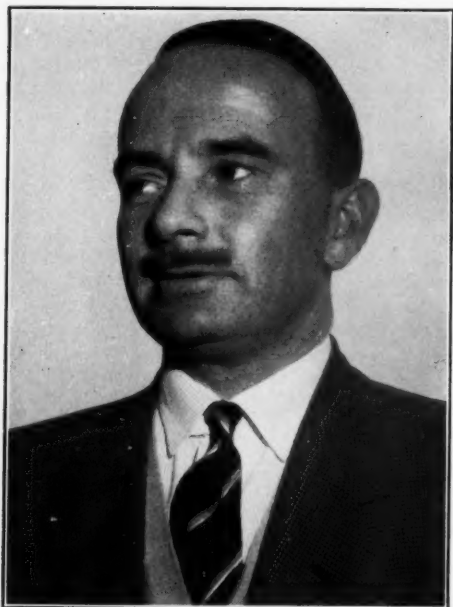
NORMAN HAMER
 A.M.C.T., F.S.D.C.

Silver Medal for valuable contributions in service to the Society



JOHN WILLIAM REIDY
 M.A.(Com.), F.R.I.C., F.T.I., F.S.D.C.

Silver Medal for valuable contributions in service to the Society



WILLIAM ROBERT LEIGH
A.M.C.T.

Bronze Medal for enthusiastic services in the
interests of the Society



JOHN RAYMENT
A.T.I., F.S.D.C.

Bronze Medal for enthusiastic services in the
interests of the Society

Mr. D. B. Moore (*General Secretary of the Textile Institute*), Mr. D. R. Balfour Park (*Clerk of the Worshipful Company of Dyers*), Mr. C. G. Hulse (*Board of Trade*), Mr. G. W. Bednall (*Editor of "The Dyer"*), Dr. J. Avery, Professor W. Bradley, Mr. G. H. Carnall, Mr. R. D. Chorley, Mr. H. Clayton, Mr. J. Ewing, Mr. T. A. Fairclough, Mr. A. D. Ferns, Mr. M. Hammond, Mr. F. A. Helme, Mr. R. J. Kerr-Muir, Mr. C. Lupton, Mr. G. B. Ratcliffe, Mr. H. Rhodes, M.P., and Mr. L. Savage; Mr. R. J. Hannay (*President-elect of the Society*), Mr. L. E. Jones (*Honorary Patent Agent*), Mr. Clifford Paine (*Honorary Member and Past President*), Dr. C. J. T. Cronshaw (*Perkin Medallist, Honorary Member, and Past President*), Mr. J. T. Marsh (*Perkin Medallist*), Dr. H. Ris (*Gold Medallist and Vice-president of the International Federation of Associations of Textile Chemists and Colourists*), Mr. I. D. Rattee and Dr. W. E. Stephen (*Gold Medallists*); Mr. H. F. à Brassard, Mr. G. G. Bradshaw, and Mr. J. W. Reidy (*Silver Medallists*); Mr. W. R. Leigh and Mr. J. Rayment (*Bronze Medallists*).

The following officers and other leading members of the Society also were present—Mr. H. H. Bowen and Mr. F. Scholefield (*Honorary Members and Past Presidents*); Mr. John Barritt (*Honorary Member and Vice-president*); Mr. R. C. Oakley, Mr. W. Penn, Dr. T. Vickerstaff, and Mr. G. S. J. White (*Vice-presidents*); Mr. A. Waddington (*Honorary Treasurer*); Mr. G. B. Angus, Mr. A. W. Carpenter, Mr. S. R. Cockett, Mr. R. K. Fourness, Mr. H. Hampson, and Mr. J. G. Hopkinson (*Ordinary Members of Council*); Mr. F. Attack, Mr. G. H. Binns, Mr. A. S. Cluley, Mr. J. N. Littler, Mr. K. Meldrum, Mr. J. G. B. McCallum, Mr. J. Porter, Mr. J. Rankin, Miss Esmée Smith, Mr. H. W. Taylor, Mr. H. Turner, Mr. H. A. Turner, Mr. W. J. Watts, and Mr. R. Woods (*ex-officio Members of Council*); Mr. B. C. Burdett, Mr. W. R. Hermes, and Mr. A. S. Fern (*Junior Branch Officers*); Mr. A. Thomson (*Chairman of the Publications Committee*); Mr. J. V. Summersgill (*Honorary Secretary of the Publications Committee*); Mr. K. McLaren (*Chairman of the Fastness Tests Co-ordinating Committee*), Mr. C. L. Bird and Mr. C. O. Clark (*Joint Acting Editors*).

The total number of diners was 607, a record in the history of the Society.

"THE SOCIETY OF DYERS AND COLOURISTS"

After the toast of "Her Majesty the Queen", that of "The Society of Dyers and Colourists" was proposed by—

Sir DONALD KABERRY, Bt., T.D., M.P., who said that he had the honour of proposing it to a gathering of tinctorial technologists who appeared somewhat to belie the colours of their profession; indeed, so far as he could see, they appeared garbed as if to say farewell to Leeds United from the First Division. But, dull though their appearances might be, he knew that good, red—perhaps even purple—blood would flow quickly in their veins when he coupled the toast with the name of their President, Mr. Malcolm Stevenson, who presided garbed in the new authority with which

he had been invested earlier that day, and to whom they offered salutations and good wishes for his term of office. He would add lustre to the high and dignified position he now adorned. The President was a very wise man indeed: he had had the good sense to come to Leeds for his professional training.

Might he, too, offer a word of congratulation and say "Well done!" to the Immediate Past President, Mr. John Boulton. All members of the Society would wish to pay tribute to his devotion to his duties as President. They would, too, ever remember his triumph at the International Congress in London last year.

He thanked the General Secretary for having briefed him so well in his duties that night. Mr. Nicholls had supplied him with full data of the Society from the date of its foundation in 1884. It was abundantly clear from the Society's records that its members covered all those activities associated with the use of colour in every sphere of modern life.

Each age made its contribution to the sum of knowledge, if not always to the sum of human wisdom. The advance—sometimes rapid—in the standard of life in Britain within the last half-century had been brought about more by the efforts of scientists, technologists and industrialists than by the politicians at Westminster. Politicians imagined that they could distribute wealth, but they were usually not very expert at creating wealth. Indeed, Government interference was not always successful, whether one considered it as starting from King Alfred's care of the cakes or at some later stage in history. Progress depended upon the efficiency and enthusiasm with which everybody played their part. The discovery of new chemical processes and their development and application for industrial and commercial purposes were bringing about a revolution in the standard of life as striking as anything ever known. It was important, if Britain was to survive and its people were to continue to have a rising standard of living, that it should be in the forefront of all technical and commercial development. There was a very old Chinese proverb which said: "If people take no care for the future, they will soon have to sorrow for the present".

He had looked most inquiringly for any note of colour which could be introduced in a speech to their great Society. It so happened that, 191 years ago that day, the Duke of Wellington was born. The Duke had introduced the "Thin Red Line"; he had also described his soldiery in terms denied to the imagination of many who today sought names for new colours. Last Tuesday, the Finance Bill had been published: it said good-bye to "the golden handshake". In its place had been substituted "the silver slap on the shoulder"—there was still a bit left for "good boys".

It was fitting and proper that once again the Society should have its Annual General Meeting and Dinner in the great City of Leeds, which was so closely associated with many of the technologies with which the Society's members dealt daily.

The University of Leeds, with its great departments—household names, known to all—had on its staff members of the Society who had made great contributions to the dyeing and textile industries throughout the world. The Wool Industries Research Association also, Leeds could claim, had achieved world-wide fame.

Sir Donald said that we owed very much to colour—gay or sad, passive or active. Perhaps colour was accepted too easily. What was clear was that, in today's crowded life, more and more colour was needed in towns, houses, clothing and all the things we used. Much had already been achieved. Homes and furnishings had been brightened by attractive colour, whilst the personal adornments of many of our fellow beings created constantly a feeling of utter wonder.

When the Society was first formed, in 1884, the country had been worried about the Egyptian problem. For six months no news had been heard of Gordon in Khartoum. A man called H. M. Stanley had arrived in Britain from the Congo. A Democratic Committee for the Abolition of the House of Peers had been created—it had since been dissolved. There had been great apprehension in regard to competition from a then recently united German Empire in the centre of Europe, and trading fears arising from a vastly increased population in the United States of America.

A parallel could, perhaps, be drawn with some of the present-day fears—the problems of the Six and the Seven. All hoped for some form of closer integration, but there was a fear that in order to attain it, the dyestuffs industry might be called upon to pay too high a price. Really what was wanted was a greater volume of trade. There were vast areas of the world still not fully developed, and he expressed the wish, in proposing the toast of "The Society of Dyers and Colourists", that its members would continue to have full orders and be able adequately and quickly to discharge them. The Society must maintain the highest educational and technical standards over the years to come. Examinations might become harder; their variety and scope would increase as the years advanced. If the country were to take the lead in technical skill and all it could bring, then the Society had a very great part to play.

He wished success to all who pursued their calling through the skill of the tinctorial arts. He asked them to promote a brighter world and to pass on their skill to those who learned at their hands; to keep their light bright as progress in the world continued, so that, in the end, the world might be flooded with colours which today we did but imagine in our dreams.

Mr. F. MALCOLM STEVENSON, J.P., M.Sc. (*President of the Society*) in responding to the toast said that, when asking Sir Donald to be his guest, he had little known what he was to bring upon himself. To ask anyone to follow Sir Donald—a member of a House where oratory abounded—was really to ask the impossible.

The President said that he had intended to describe his impressions as a "new boy" who had come

back into the affairs of the Society at the top, feeling for the first time the vigorous new growth that was taking place both at headquarters and in the Sections. Members did not realise how proud they should be of the Society to which they belonged.

He could enumerate those many facets of interest, and the impact they had upon their own and other peoples' daily lives, in order to boost members' morale, make them stick out their chests and feel proud to belong to the Society; but Sir Donald had done it for him, and he would merely appeal to members to take a legitimate and proper pride in the Society and visualise for themselves the wider horizons that lay before them.

Reference had been made to that great event last September, when the Society was host at the International Congress in London. Tribute should be paid to all those who had brought the name of the Society to the pinnacle of eminence achieved on that occasion—those who had worked in an honorary capacity; the staff; and those firms which, in the background and without publicity, had underwritten all the expense of a great operation.

Another cause of legitimate pride was that, as members had heard that afternoon, the arrangements with the Americans concerning the *Colour Index* had, after many years, been rounded off. His mind went back to February, 1946, when he had visited Canada and the United States for the first time in the post-war era. As a member of the Publications Committee he had been permitted to contact some members of the A.A.T.C.C., in order to follow up what had already been done by three members of Council and to emphasise the Society's great desire to go hand in hand with the Association in the great project. That was over fourteen years ago, and only in the last few weeks had they seen a complete rounding off of the enterprise. Such things took a long time, but it had been well worth it. Now, the Society's name stood as co-producer with the Americans of something of which its members should be inordinately proud.

He had referred to the International Congress. There were certain aspects of it which could give rise to unease. Those who had been privileged to be present must have been struck by the fact that almost all the overseas visitors had been able to converse in English with an enviable ease and facility. Few of the Society's representatives could have gone to the Continent and conversed in a native language with such fluency, facility, and speed. There was in that a germ of serious thought. These visitors were our friends, but also our competitors. We, in this country, had no prerogative or exclusive right to the good things of the world or of life. It behoved us to lose some of our insularity and to appreciate the need to go and discuss in the language of the particular country those things which were now being discovered there, and which we could neglect only at our peril.

So he would say that this became a part of the attack in the sphere of Education, in which the Society's members were increasingly playing a part.

He hoped they were proud of what was being done in that sphere. He hoped they read the *Journal* and saw what was happening in the endeavour to ensure that the young men of the future were properly trained and adequately equipped. Sir Donald had invited them to pass on to the next generation their skills and knowledge: it was a responsibility they all shouldered. Skilled educationalists had been appointed to give advice. Summer schools were being encouraged, as well as symposia, one-day conferences, and the like. It was common for schools and universities to find places in the laboratories and works scattered about the country for a few of their members during the long vacation. But not nearly as much was done as ought to be done, and he would invite those who had the power to do it, to throw open their doors far more willingly in order that young men might come in and learn for the future.

Too few university teachers had an opportunity to keep themselves up to date in modern techniques, which today went ahead at great speed. A great deal of good would result if those men, who, perforce, over the years became detached from industry, could be invited into works for short courses, in order that they might see the most modern techniques and machinery, and then go back and include them in their teaching.

If the Associateship of the Society was to be held in high esteem, it must be backed by proper status, proper standards, and proper control. Such matters inevitably required more expenditure.

Mr. Stevenson then went on to deal with the increased charge for membership, and appealed to those present to "pay up with a smile".

The Society, he said, thrived, prospered, and widened its influence by reason of the good fellowship that existed among the members in the Sections, and this should be carefully guarded and watched. The sense that everyone had a part to play should be maintained, and it was of paramount importance that members should not wait for somebody else to do all the work for them. This corporate spirit, which was their great strength and the envy of those who looked on from afar, would continue to secure their prosperity and vigour. It was a source of power for the future, and, if they held on to it, they could not go astray.

The President ended by thanking the retiring President (Mr. John Boulton), the Honorary Officers, the staff, and all those with whom he had come in contact for their kind helpfulness during his year of preparation for the Presidency. He also extended a warm welcome to the President-elect, Mr. R. J. Hannay.

HONORARY MEMBERSHIP

Mr. F. M. STEVENSON (*President of the Society*) presented a Diploma of Honorary Membership of the Society to Mr. Clifford Paine, awarded "in high appreciation of his devoted service to the Society".

PRESENTATION OF AWARDS

The President then made the following presentations and awards—

<i>Perkin Medals</i>	Dr. C. J. T. Cronshaw Mr. J. T. Marsh
<i>Gold Medals</i>	Dr. H. Ris Mr. I. D. Rattee Dr. W. E. Stephen
<i>Silver Medals</i>	Mr. H. F. à Brassard Mr. G. G. Bradshaw Mr. N. Hamer Mr. J. W. Reidy
<i>Bronze Medals</i>	Mr. W. R. Leigh Mr. J. Rayment
<i>Diplomas of Appreciation</i>	Mr. S. J. Pentecost The late Dr. A. Burawoy

Mr. Hamer was unable to be present owing to illness, and Mr. Pentecost by reason of his great age. The President said he would make personal calls on Mr. Pentecost, Mr. Hamer, and the widow of the late Dr. A. Burawoy to present their awards himself.

"OUR GUESTS"

Mr. JOHN RANKIN (*Chairman of the West Riding Section*), in proposing this toast, said that, whilst he was a firm believer in the adage "few words are best", it would be sadly remiss of him if he did not at the outset thank Council for holding the Seventy-sixth Annual General Meeting and the Annual Dinner in Leeds. This afforded him, as Chairman of the West Riding Section, the privilege and pleasure of proposing the traditional and sincere toast of "Our Guests". The interest shown in the Society's affairs was reflected not only in a record attendance of over 600, but also in the honour accorded it by the presence of so many eminent guests. They were particularly gratified to have once again as principal guest Sir Donald Kaberry, who had honoured the Society in that capacity at the Buxton Symposium in 1957. They were sorry to note there had been trouble in the House that week—about "Blue" something or other—but, as a colour-user, he had the greatest sympathy with Sir Donald, because blues were always most difficult and expensive. Lord Milner of Leeds had been their principal guest at the Annual Dinner in London in 1957. Both guests had shown themselves to be consistently true friends of the Society.

Mr. Rankin said they were particularly happy to welcome Mr. R. F. Rothwell, Prime Warden of the Worshipful Company of Dyers, and the occasion would not have been complete without the presence of Mr. D. R. Balfour Park, Clerk to the Court. Also present as guests were leaders of the colour-making and colour-using industries and of the dyeing and finishing industries, as well as manufacturers of cloth, members of the clothing and distributive trades, and learned gentlemen from Leeds and other universities and institutions of technology. Kindred, honoured societies were represented strongly, although he would mention only two names: Sir William Garrett, Chairman of the Association of British Chemical Manufacturers, and their staunch overseas friend and Gold Medallist, Dr. Heinrich Ris, Vice-President of the International Federation of Associations of Textile Chemists and Colourists. And lastly, but by no

means least, the guests included those people who were the very sinews of the Society—the Honorary Secretaries of Sections.

The Right Honourable Lord MILNER OF LEEDS, P.C., M.C., T.D., D.L., LL.B., Hon.L.L.D., in responding to the toast, said that he was very greatly honoured in having been invited once again to the Society's Annual Dinner and in having the opportunity of responding, on behalf of the guests, and thanking the Society for its generous hospitality and the very happy company in which they found themselves.

Unlike the celebrated judge who complained that he had been asked to make a speech, when all he was accustomed to doing was delivering a few sentences, he was greatly relieved at the very kind references that Mr. Rankin had made to the guests that night.

As a simple lawyer, he knew very little of the work of dyers and colourists, and he imagined that applied to quite a number of the guests, but one marvelled at the miracles they undoubtedly worked. Their products, infinite in their variety,

fastness and applicability to so many different materials, were really very remarkable and a great tribute to the industry and those who worked in it.

He did not know for what they really were responsible. He was quite certain they were not responsible for the "golden handshake" or the "Blue Streak"—Sir Donald must take due responsibility for that.

He paid tribute to their industry. It could render a service, in present circumstances, in very many different directions. The vital need was to export, and the industry played, and could play, a great part in furthering the export trade of the country. There were some who did not, perhaps, pay to the export trade the attention which they might, and who would render a great service to the country if they made greater efforts in that direction. He would make a plea that that vital need be met, in so far as the industry could meet it.

He thanked the Society, on behalf of its guests, for a delightful evening, and he expressed the hope that the Society would continue to prosper and that its members would prosper with it.

COMMUNICATIONS

The Chemistry of Esters of Leuco Vat Dyes IV—Oxidation with Solutions of Nitrous Acid

A. JOHNSON and A. P. LOCKETT

When the sulphuric esters of leuco vat dyes are developed with acidic nitrous acid solutions in the complete absence of oxygen, the sulphate groups are removed only by hydrolysis; the resulting leuco dye is then oxidised by nitrous acid. When oxygen is present development is far more rapid, the majority of sulphate groups being removed by direct oxidation with hydroxyl radicals *via* a chain mechanism. The chain reaction is initiated by hydrogen peroxide (or HO_2 radical) and propagated by the homolytic fission of pernitrous acid and its re-formation from nitrogen dioxide and nitrous acid. This mechanism is thought to operate in normal development. A modification of the technical development process is suggested.

Introduction

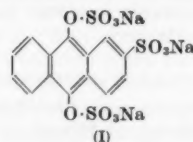
Earlier work in this series has been concerned with the development of sulphuric esters of leuco vat dyes (vat esters) by means of acidic hydrogen peroxide, and it has been shown that direct oxidation of the ester groups occurs by means of hydroxyl radicals^{1,2}. Such behaviour must be anticipated in any vat ester system in which oxidising radicals are produced, and probably occurs in all technical development processes. An important technical process is the development with sodium nitrite and mineral acid solutions, i.e. with nitrous acid, and it was felt that a detailed investigation of this process would be rewarding.

Some reactions with nitrous acid were carried out in the earlier work¹, and it was found that oxygen dissolved in the reaction solutions had a profound effect, an increase in the concentration of oxygen leading to a considerable increase in the rate of development. This effect is obviously important in dyeing and printing, but is a complication when investigating a reaction mechanism.

It was therefore thought desirable to establish the course of the reaction between a vat ester and nitrous acid in the complete absence of oxygen before turning to development when oxygen is present. It is not possible to remove oxygen completely from aqueous solutions of the reactants by physical means, and the use of reducing agents

for this purpose was considered inadvisable, since they would also react with nitrous acid. Fortunately, the earlier experiments with nitrous acid had indicated that residual oxygen in the solutions reacts rapidly and is completely consumed, so a study of the later stages of the complete development should enable the course of the reaction in the absence of oxygen to be determined.

Accordingly, the vat ester of anthraquinone-2-sulphonic acid (I), which had been used as a model compound before, was allowed to react under controlled conditions with acidic solutions of sodium nitrite from which as much oxygen as possible had been removed, and the course of the reaction was followed. Later, the effect of varying the oxygen concentration was also studied.



Experimental

MATERIALS

The vat ester was prepared from anthraquinone-2-sulphonic acid as described previously¹. Other materials were of AnalaR quality.

METHOD

The experiments were carried out in the apparatus described in Part I¹ for use with a nitrogen atmosphere. A supply of hydrochloric acid sufficient for several experiments was first de-aerated as completely as possible by repeatedly freezing and evacuating in a separate flask. The vacuum in this flask was finally released by means of Wembley nitrogen (O_2 content 10 p.p.m.). Water was similarly treated in a second flask.

In order to carry out an experiment, the reaction vessel was first flushed with nitrogen for 30 min., during which time a weighed amount of solid vat ester was introduced into one limb in a glass thimble. An appropriate volume (usually 74 ml.) of water was then transferred into this limb from the storage flask, under a nitrogen atmosphere, and also the sodium nitrite in 1 ml. of a nitrogen-saturated solution. Hydrochloric acid was transferred to the second limb of the vessel and flushing with nitrogen continued for a further 15 min. before starting the reaction by inverting the vessel to mix the solutions. The reaction was followed by periodically running out aliquot portions for analysis as below.

*Analytical Quinone*¹

This was determined as before by adding 5 ml. of reaction solution to a 5% solution of sodium hydrosulphite in N. sodium hydroxide and measuring the optical density of the resulting quinolate ion at 4300 Å. In Part I¹ it was reasoned that all compounds derived from the vat ester were reduced to the quinol, so that the concentration measured represented the total vat ester which had reacted, and not merely that which was completely oxidised to quinone. The opportunity was taken to confirm this supposition by the use of a new method for analysing a mixed spectrum put forward by Dewar³. This showed that the alkaline solution used for absorptiometry did in fact contain only quinol and unchanged vat ester; it also showed that the acidic reaction solution (before reduction) contained other species in addition to unchanged vat ester and quinone.

Nitrous Acid

The method of Shinn⁴ was used, in which sulphanilamide is diazotised and then coupled with *N*-(1-naphthyl)ethylenediamine. Reaction solution (5 ml.) was added to a 0.5% solution (2 ml.) of sulphanilamide in 50% hydrochloric acid, followed after 5 min. by a 0.1% aqueous solution of *N*-(1-naphthyl)ethylenediamine hydrochloride (1 ml.) and the volume made up to 25 ml. with water. The optical density of the reddish-violet solution was measured at 5350 Å. The colour is stable for 3 hr.

Sulphate Ions

In the work with hydrogen peroxide much useful information was obtained by measuring the concentration of sulphate ions in the acidic oxidising solution, as well as after making the solution alkaline. Unfortunately this could not be done in the present work, because much less

sulphate was liberated. Attempts to improve the sensitivity of the analysis were not successful, but it was possible to show that, as before, additional sulphate was liberated when a partly reacted solution was made alkaline.

All experiments were carried out at 25°C. in 0.1 N. hydrochloric acid. The vat ester concentration was 10^{-3} M. for the main series of experiments and the nitrous acid concentration was varied between zero (for hydrolysis measurements) and 2.5×10^{-4} M. These conditions were found to give reasonable reaction rates, and the excess of vat ester ensured that in most cases its concentration did not change significantly during an experiment. The concentration of oxygen was not known, but was constant from one experiment to another and was very low. Light was always excluded.

Results and Discussion

The first problem was to determine the course of the reaction during its later stages, when all the dissolved oxygen had been consumed. The solution to this problem came quite readily when the rates of reaction were measured at various concentrations of nitrous acid and compared with the rate in the absence of nitrous acid. This last rate is termed the rate of "hydrolysis", since no oxidising agent was deliberately added, but a small amount of oxidation will occur here due to the residual oxygen. However, this also is confined to the early part of the reaction.

The rate-of-oxidation curves always lie above the hydrolysis curve, i.e. some vat ester is decomposed by oxidation in excess of that which is hydrolysed. However, after a certain time of reaction the oxidation curves become parallel to each other and also to the hydrolysis curve. Some examples are illustrated in Fig. 1. It is seen that the three curves where nitrous acid is present become parallel after ca. 120 min., and also

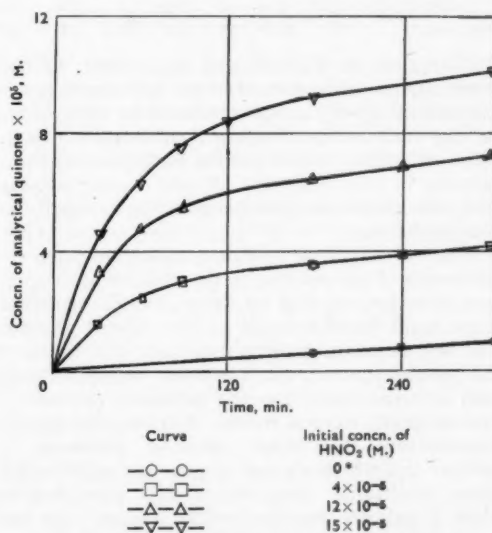
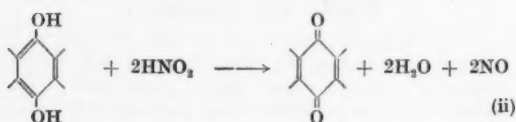
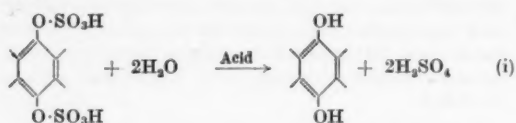


FIG. 1—Effect of Nitrous Acid on Reaction Rate (Initial concn. of vat ester 10^{-3} M.)

parallel to the hydrolysis curve obtained in the absence of nitrous acid. Similar results were obtained for other vat ester concentrations.

The result in Fig. 1 can only mean that, after ca. 120 min., the same reaction rate is being measured in each case, viz. the rate of hydrolysis. Thus, in this region no vat ester can be undergoing direct oxidation, and the only reaction which consumes vat ester is hydrolysis. In this region, then, the simple two-stage mechanism of (i) hydrolysis to the leuco compound followed by (ii) oxidation of this to the parent quinone actually occurs.



This supposition can be checked from the rates of consumption of nitrous acid because, according to (ii), two molecules of nitrous acid should be consumed for each molecule of quinone produced. In Table I are given the rates of consumption of nitrous acid during the hydrolysis-dependent part of the reaction for various initial concentrations. It is seen that the rate increases with concentration to a constant value which is double the rate of formation of analytical quinone, as demanded by (ii).

TABLE I

Rate Constants for Consumption of Nitrous Acid (*k*) at Various Concentrations

(Vat ester concentration = 10^{-3} M.; rate constant for vat ester = 19×10^{-4} min. $^{-1}$)

Initial $[\text{HNO}_2]$ $\times 10^3$, M. ...	2	4	6	8	15	20	25
$10^4 k$, min. $^{-1}$...	33	35.5	35	36	40.2	42	42

The values in Table I also imply that, at the lower nitrous acid concentrations, the quinol is not oxidised as rapidly as it is produced by hydrolysis, i.e. free quinol accumulates in the reaction solution. (This, of course, would not be distinguished from quinone in the analysis.) At the higher nitrous acid concentrations, quinol is removed as rapidly as it is produced.

It is felt that these results establish conclusively the course of the reaction in the absence of oxygen, and attention can now be directed to the region of more rapid reaction when oxygen is still present. The first requirement is to establish with which of the species present, viz. vat ester, quinol, nitrous acid or nitric oxide (i.e. the reduction product of nitrous acid), oxygen reacts. Nitrous acid can be immediately discounted, because solutions of nitrous acid which contain oxygen are stable under these conditions. Also, the results presented in Part I indicate that molecular oxygen will not react with the vat ester; hence the choice lies between quinol (produced by hydrolysis) and nitric oxide (from reduction of nitrous acid).

The results in Fig. 1 are an important clue. It is seen that the duration of the rapid part of the reaction is constant, irrespective of the nitrous acid concentration, and hence of the nitric oxide concentration. Since the initial concentration of oxygen was the same in each experiment, it follows that, for a constant concentration of vat ester, oxygen is always consumed at a constant rate. At higher vat ester concentrations the duration of the oxygen-affected reaction diminishes (see Table II), but remains independent of the nitrous acid concentration.

TABLE II

Effect of Vat Ester Concentration on Duration of Oxygen-affected Reaction

Vat ester concentration $\times 10^3$, M. ...	4.0	2.0	1.0	0.5
Duration, min. ...	40-50	80-90	110-120	180

These observations are all in accord with a reaction between oxygen and quinol because, for any vat ester concentration, the production of quinol by hydrolysis is independent of nitrous acid. Confirmation can be obtained by adding oxygen to reaction solutions after the reaction rate has become equal to the rate of hydrolysis, firstly when the concentration of nitrous acid is too low to remove quinol as rapidly as it is produced, and secondly when it is sufficiently high (see Table I). In the first case free quinol is present in the solution, so the introduction of oxygen should cause a marked increase in rate if oxygen reacts with quinol. In the second case the increase in rate would be delayed, because no free quinol would be present. On the other hand, if oxygen reacts with nitric oxide there should be a greater increase in the second case, because the accumulation of nitric oxide is there the greatest. The results of this experiment (Fig. 2), in which oxygen was added by injecting 5 ml. of oxygen-saturated water through the draw-off tap of the vessel,

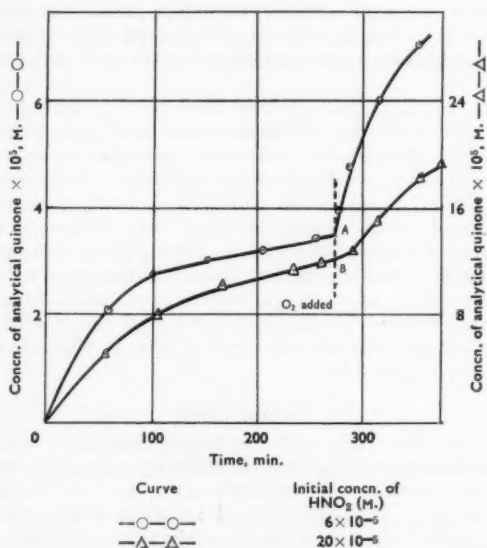


FIG. 2—Effect of Adding Oxygen

establish clearly that oxygen reacts with quinol rather than with nitric oxide, since the rate after point *A* (quinol present) is greater than after point *B* (no quinol) and also greater than the original rate, whereas the rate after *B* is lower than the original rate.

Attention was next directed towards the effect of variations in the conditions on the first part of the reaction, and the concentrations of nitrous acid, vat ester, and oxygen were varied in turn. Variation

in oxygen concentration was effected by modifying the de-oxygenation process. The results of these variations are illustrated in Fig. 3, 4, and 5. Fig. 3 shows that increasing the nitrous acid concentration causes an increase in rate. At the same time the duration of the rapid reaction remains at 120 min. The result of varying the oxygen concentration, however, is the reverse of this (Fig. 4). The rate of the rapid reaction remains unchanged, but its duration increases with increasing oxygen concentration. Finally, changing the concentration of vat ester has the effect of increasing the rate but shortening the duration of the rapid reaction (Fig. 5).

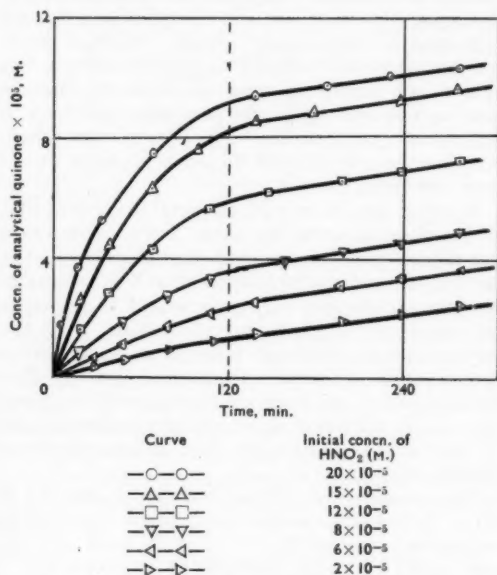


Fig. 3—Effect of Varying the Concentration of Nitrous Acid (initial vat ester concn. 10^{-3} M.)

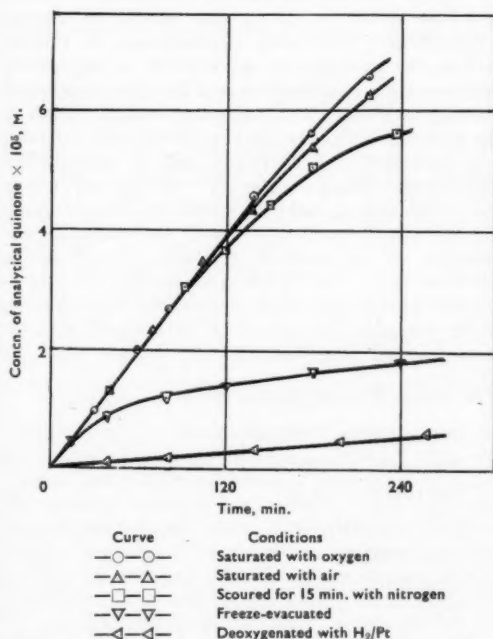


Fig. 4—Effect of Varying Oxygen Concentration (vat ester initially 10^{-3} M. and nitrous acid 2×10^{-5} M.)

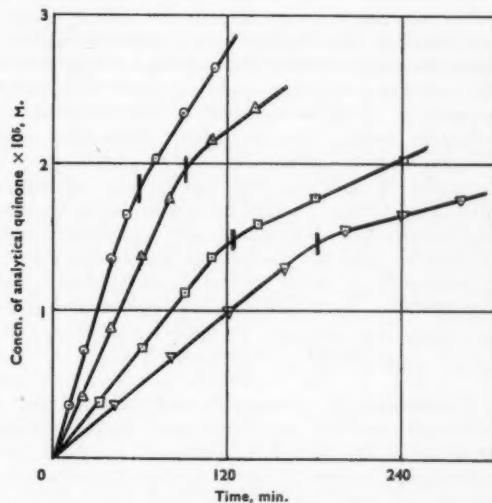
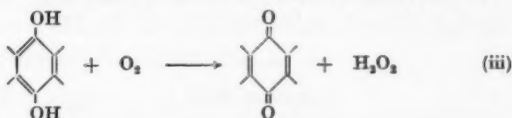


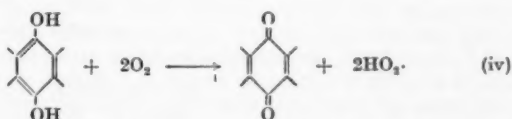
Fig. 5—Effect of Varying Vat Ester Concentration (initial concn. of nitrous acid 2×10^{-5} M.)

All these observations again support the suggestion that oxygen is consumed by reaction with the quinol produced by hydrolysis. At higher vat ester concentrations, hydrolysis proceeds more rapidly and hence oxygen is consumed more rapidly.

Autoxidation of the quinol leads to the formation of hydrogen peroxide *via* the hydroperoxy radical—



or



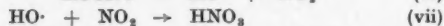
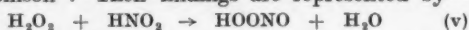
One (or both) of these species must then react with nitrous acid and thereby induce direct

oxidation of the vat ester, by a mechanism which will also consume nitrous acid and whose rate must be controlled by the concentration of nitrous acid. Furthermore, this mechanism must include a chain reaction, since the rate of oxidation can be many times greater than hydrolysis, i.e. one (or two) molecules of oxygen brought into reaction by means of quinol can cause the oxidation of several molecules of vat ester (Table III).

TABLE III
Ratio of Oxidation to Hydrolysis
(Vat ester concentration = 10^{-3} M.)

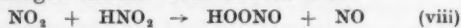
10^5 [HNO ₂], moles/litre ...	2	4	8	15	25
Oxidation: hydrolysis ratio	10.7	26	28.5	67	86

The reaction between nitrous acid and hydrogen peroxide has been studied by a number of workers. Thus Raschig in 1904⁵ showed that a mixture of the two was a stronger oxidising agent than either separately. This he ascribed to the formation of pernitric acid, but in 1910, Schmidlin and Massini⁶ stated that the active entity was a peroxide of nitrous acid having the structure HOONO. Other workers have from time to time suggested the formation of pernitrous acid (HOONO), and the compound has recently been investigated in some detail by Halfpenny and Robinson⁷. Their findings are represented by—



Pernitrous acid is unstable and splits to give a hydroxyl radical and nitrogen dioxide. The activity of the mixed solutions of peroxide and nitrous acid is due to these species; e.g. aromatic compounds can be hydroxylated and nitrated, methyl acrylate is polymerised, etc. In the absence of a suitable substrate, the radicals recombine to give nitric acid.

Thus, if hydrogen peroxide is produced in a vat ester solution as suggested above, pernitrous acid will be formed and could be responsible for the observed enhanced oxidation. Since it is known¹ that hydroxyl radicals can cause direct oxidation of a vat ester, this supposition appears reasonably likely. However, the direct oxidation of the vat ester is a chain reaction, so that a chain-carrier is necessary in order that pernitrous acid can be re-formed. We suggest that this occurs by reaction of nitrogen dioxide with nitrous acid—



A complete reaction mechanism can now be given to explain all the observed effects, viz.—

Reactions (1) and (2) have been established and (3) and (4) have been shown to occur by Halfpenny and Robinson; 2(a) and 3(a) are alternatives to (2) and (3). The oxidation of the vat ester, reaction (5), has also been shown to occur¹. Reaction (6) is proposed as a chain-propagating reaction, so that (4), (5), and (6) together provide a chain mechanism for the oxidation of the vat ester. Reaction (7) acts as the chain-stopper.

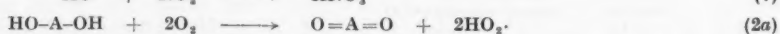
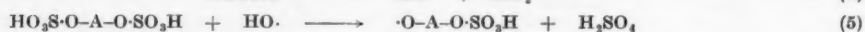
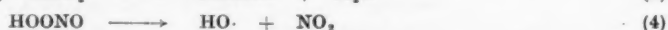
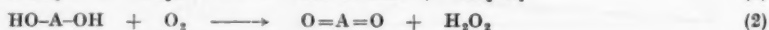
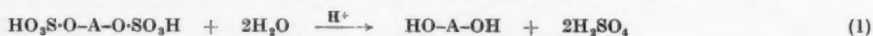
A novel feature of the proposed scheme is that nitrous acid can be involved in a reaction chain leading to the oxidation of a substrate and its own reduction to nitric oxide. Such a reaction chain was not observed by Halfpenny and Robinson, but this is not surprising since in their experiments nitrous acid and hydrogen peroxide were used in equivalent proportions. To obtain the chain reaction the nitrous acid should be in large excess over the peroxide.

We were now faced with the need to confirm that such a chain reaction can occur. For this purpose a vat ester is not the most suitable substrate to use, since it can undergo hydrolysis as well as oxidation. A more satisfactory substrate would be one that can react only with hydroxyl radicals, from the various species which are believed to be present in the reaction solution. It is well known that acrylonitrile is polymerised by hydroxyl radicals, but not by nitrous acid, or nitrogen oxides (which in fact act as inhibitors), and is accordingly a suitable substrate.

Two solutions of nitrous acid (10×10^{-5} M.) in 0.1 N. hydrochloric acid were prepared, one of them containing acrylonitrile (1.5 M.). To each solution was added hydrogen peroxide (4×10^{-5} M.).

The concentration of nitrous acid fell rapidly in each case, and then became constant at 6.0×10^{-5} M. for the solution not containing acrylonitrile and 1.95×10^{-5} M. for the one which did contain acrylonitrile. The extra consumption of nitrous acid in the presence of acrylonitrile is supporting evidence for a chain reaction of the type suggested. Further evidence that the same chain occurs in the reaction with acrylonitrile as with the vat ester was provided by carrying out a competition experiment. The vat ester (10^{-3} M.) was allowed to react with nitrous acid (6×10^{-5} M.), firstly under the standard conditions and secondly in the presence of acrylonitrile (0.8 M.). The resulting reaction rates are illustrated in Fig. 6. It is evident that acrylonitrile is oxidised in preference to the vat ester: the rate of vat-ester reaction in the presence of acrylonitrile is almost that of

REACTIONS OCCURRING IN THE SYSTEM VAT ESTER-NITROUS ACID-OXYGEN



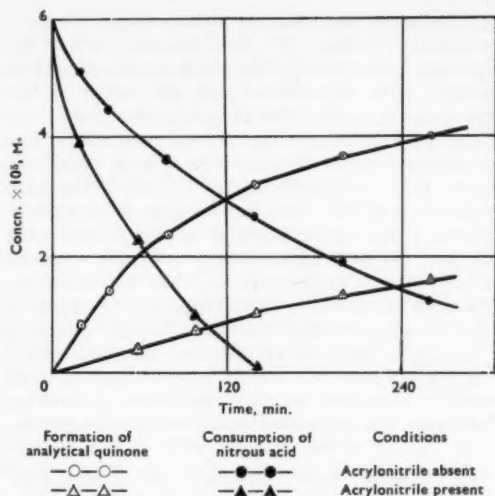


FIG. 6—Effect of Addition of Acrylonitrile (0.8 M.) to Vat Ester (10^{-3} M.) and Nitrous Acid ($6 \times 10^{-3} \text{ M.}$)

hydrolysis, this providing quinol and hence hydrogen peroxide to initiate the chain mechanism which consumes nitrous acid and acrylonitrile.

Finally, confirmation that the chain reaction is initiated by hydrogen peroxide can be sought by adding hydrogen peroxide to a solution of vat ester and nitrous acid. An increased production of analytical quinone, in excess of the peroxide added, would be expected. That this is so is shown clearly in Fig. 7.

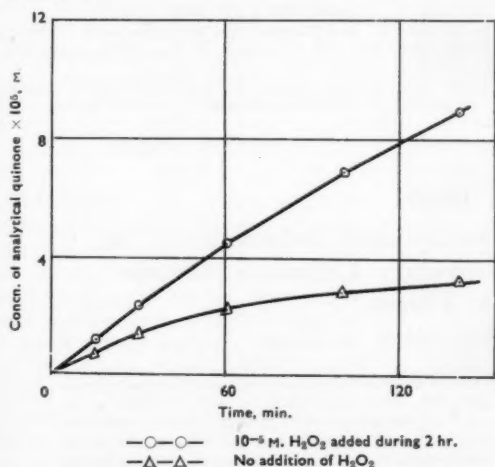
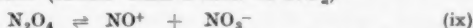


FIG. 7—Effect of Addition of Hydrogen Peroxide during Development (vat ester initially 10^{-3} M. and nitrous acid $6 \times 10^{-3} \text{ M.}$)

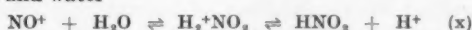
These various observations all lend support to the suggested overall reaction scheme. There is independent evidence for each individual step in this scheme except for reaction (6), i.e. the production of pernitrous acid from nitrous acid and nitrogen dioxide. If this reaction occurs, then the addition of nitrogen dioxide to a solution of vat ester and nitrous acid should lead to an accelerated rate of consumption of both reactants. Unfortunately nitrogen dioxide readily dimerises, so it is not possible to make a direct addition of the

monomeric form. However, a solution of Cibantine Brown G (C.I. Solubilised Vat Brown 5) and nitrous acid was exposed briefly to the fumes from liquid dinitrogen tetroxide. The vat dye was certainly produced more rapidly than in a similar solution unexposed, but the concentration of nitrous acid rose instead of falling.

The explanation of this apparent anomaly can be deduced from consideration of the molecular structure of nitrogen dioxide and its dimer, which have recently been reviewed in some detail by Gray⁸. The dimer (N_2O_4) is believed to exist in aqueous solution in equilibrium with nitrosonium nitrate (rather than 2 mol. of NO_2)—

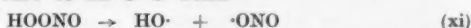


This immediately leads to the formation of nitrous acid from the known reaction between nitrosonium ion and water—

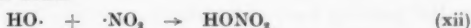


thus accounting for the well-known observation that the dissolution of dinitrogen tetroxide in water produces equimolar quantities of nitric and nitrous acids. These reactions readily account for the experimental observation with Cibantine Brown G. However, in a normal vat ester development, monomeric nitrogen dioxide is present only in low concentration in the presence of a much higher concentration of nitrous acid, so the proposed reaction with nitrous acid is much more likely than dimerisation.

The fission of pernitrous acid must involve the rupture of an O-O bond—



whereas the recombination of $\text{HO}\cdot$ and NO_2 to form nitric acid involves the formation of an O-N bond—



i.e. the structure of nitrogen dioxide must change from $\cdot\text{O}-\text{N}=\text{O}$ to $\text{O}=\text{N}=\text{O}\cdot$. In the proposed reaction scheme (p. 416), it is an implicit assumption that nitrogen dioxide reacts with nitrous acid to form pernitrous acid before this electronic change takes place—



(This is equivalent to saying that the electronic transition is the chain-stopping reaction.) It also appears likely that the stable monomeric form of nitrogen dioxide will have the structure $\cdot\text{NO}_2$. In that case, it could not react with nitrous acid to produce pernitrous acid and its addition to a vat ester-nitrous acid solution would not be a test for the reaction mechanism.

Conclusions

An important feature of this investigation has been the demonstration that nitrous acid can bring about the oxidation of various substrates via a chain mechanism. It is felt that this may be a general property of nitrous acid if the system is one in which hydrogen peroxide (or $\text{HO}_2\cdot$) can be produced and hence cause the initial formation of pernitrous acid. Hydrogen peroxide (or $\text{HO}_2\cdot$) is likely to occur if oxidation of the substrate is a two-electron process, as is most common for

organic compounds, the intermediate radical reacting with oxygen.

The results obtained in this investigation are of value in relation to the technical application of vat esters by the nitrite method. Under the usual conditions a plentiful supply of oxygen is assured, since the solutions would normally be saturated with air, and also because the cloth is often "skied" after its passage through the developing bath. It can now be seen that this access of air is a fundamental part of development, and that if air were excluded the nitrite process would be unusable. In the usual recipes for nitrite development a large excess of sodium nitrite is used (typical figures from manufacturers' literature show that in some cases a hundredfold excess is used). The purpose of this can now be seen—it is simply to favour reaction (6) (i.e. chain propagation) at the expense of reaction (7) (chain-stopping). This in turn results in longer reaction chains and a greater yield of vat dye. A similar effect could be achieved if the number of chains was increased, i.e. if reaction (3) could be promoted, and this can most simply be done by adding hydrogen peroxide (see Fig. 7). We therefore suggest that the addition of hydrogen peroxide to a nitrite development bath may be of value, particularly for those vat esters which are known to be slow developers (e.g. Cibantine Brown G and Indigosol Red 12B (C.I. Solubilised Vat Red 8)). The amount of sodium nitrite needed can then be reduced.

This effect was clearly demonstrated for the development of a large number of commercial vat esters in solution. Laboratory tests with the above-mentioned vat esters, applied to a viscose rayon cloth by padding, have shown that the inclusion in the padding bath of hydrogen peroxide equal to

1% (molar) of the sodium nitrite brings about a noticeable increase in development rate. An important point here is that the peroxide should be included with the nitrite and vat ester in the impregnating bath. The inclusion of nitrite only in the impregnating bath and of peroxide in the developing bath is not satisfactory and leads to a slower rate of development. This is because hydrogen peroxide then has to diffuse through a layer of nitrous acid before it can reach the cloth and the dye and, in so doing, it serves only to oxidise nitrous acid to nitric acid. Obviously, the behaviour of vat esters on cloth cannot be expected to be an accurate reproduction of their behaviour in a simple solution. Extensive trials have not been carried out, and clearly a great deal of work would be necessary to ascertain the best working conditions, but such work could well be rewarding.

* * *

We express our gratitude to Courtaulds' Educational and Scientific Trust Fund and to the Salters' Institute of Industrial Chemistry for awards which enabled one of us (A.P.L.) to take part in this work.

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Quinonoid Dyes

XIV*—Relation between Coplanarity and Substantivity of Quinonoid Dyes applied to Secondary Cellulose Acetate and Cellulosic Fibres

E. H. DARUWALLA, S. S. RAO, and B. D. TILAK

Two series of coplanar dyes derived from naphthaquinones have been synthesised, and for each dye a corresponding non-coplanar compound has also been obtained. Dyes belonging to one of these series (I–X) could be applied to secondary cellulose acetate rayon from aqueous dispersion, whilst cellulosic fibres could be dyed satisfactorily from an alkaline reducing bath with dyes belonging to the second series (XI–XX). The affinity values of these dyes have been determined, and the importance of coplanarity for substantivity is discussed.

INTRODUCTION

One of the requirements for a direct cotton dye—if it is to show affinity for cellulose—is that the aromatic nuclei in the dye molecule should be capable of assuming a coplanar configuration. Hodgson and Marsden¹ found support for this hypothesis from the examination of substituted benzidine derivatives. Venkataraman², however, has quoted instances where non-coplanar dyes have been shown to possess substantivity. Since,

in these studies, no quantitative determination of substantivity was made, the conclusions derived from them need confirmation. With vat dyes the evidence to support the requirement of coplanarity for substantivity is not conclusive, and examples cited in the literature are not very satisfactory. Lukin³ observed that, whereas 1,1'- and 1,2'-dianthraquinonylamine were substantive, 2,2'-dimethyl-1,1'-dianthraquinonylamine was non-substantive. This lack of substantivity was attributed to the

* Part XIII was published in *J. Sci. Ind. Res. (India)*, **17B**, 483 (1958).

non-coplanar character of the dye. Peters and Sumner⁴ showed that substitution into the *ortho* position of the benzene ring of 1-benzamido-, and 1,4- and 1,5-dibenzamido-anthraquinones greatly reduced the affinity of these dyes for cellulose. They attributed this reduction in affinity to the inability of the dye molecule to attain a planar configuration due to *ortho* substituents. A better explanation for this decrease in affinity was suggested by Valentine⁵, viz. the possibility of internal chelation of the amide group to the *ortho* substituent, resulting in a decrease in hydrogen bonding between the amide group and the cellulose. The order of decreasing affinity with the *ortho* substituents agreed much better with their capacity for chelation than with that to be expected purely on the basis of non-coplanarity.

With disperse dyes on secondary cellulose acetate, no work has been reported which suggests that planarity of the dye molecule promotes substantivity.

The present work was undertaken in order to study the effect of coplanarity on substantivity, using selected quinonoid dyes and secondary cellulose acetate and cellulosic fibres. A preliminary report on the synthesis of some of these dyes and their non-coplanar analogues has been published by Tilak and Rao⁶.

EXPERIMENTAL

Dyes

In Table I are shown pairs of quinonoid dyes. One dye of each pair is coplanar, whilst the other is the corresponding non-coplanar compound, as is seen by constructing molecular models, using Courtaulds' type of atomic models, which allow for the valency strain. Fig. 1 and 2 illustrate molecular models for the coplanar (I) and non-coplanar (II) disperse dyes, whilst Fig. 3 and 4 show models of coplanar (XIII) and non-coplanar (XIV) vat dyes. The carbon atoms in the quinonoid ring to which the anthracene residue is attached are shown as tetrahedral carbon atoms, although the chemical evidence⁷⁻⁹ strongly supports the benzoquinone structure shown in Table I.

References¹⁰⁻¹⁹ for the methods of synthesis are given in Table I for the planar dyes; methods of synthesis of the non-coplanar compounds will be published by Tilak and Rao^{6, 20}.

Dyeing Procedure

Bright secondary cellulose acetate yarn was desized by extraction with petroleum ether at

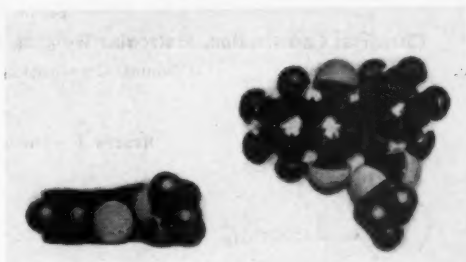


FIG. 1—Coplanar Disperse Dye (I)

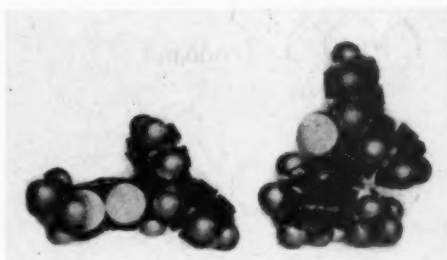


FIG. 2—Non-coplanar Disperse Dye (II)

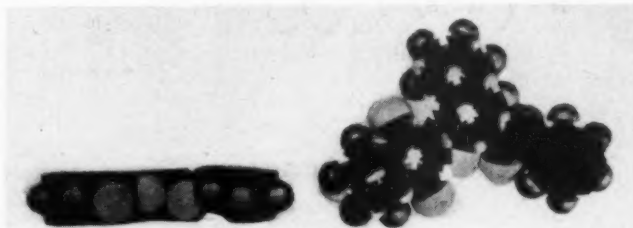


FIG. 3—Coplanar Vat Dye (XIII)

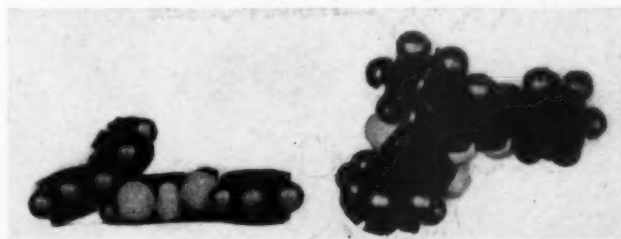


FIG. 4—Non-coplanar Vat Dye (XIV)

40–60°C. for 24 hr. Fine suspensions of dyes I–X were prepared from purified crystalline materials by grinding with distilled water for 48 hr. in the apparatus described by Daruwalla and Limaye²¹. Stock solutions of the dispersions were prepared, and the requisite quantities of stock solution were used for dyeing. Dyeing was carried out in 100-ml. round-bottom Pyrex flasks fitted with a condenser to prevent loss of liquor through evaporation. A loop-type stirrer passing through the condenser and rotating at 240 r.p.m. provided satisfactory agitation. In all experiments with secondary acetate, 0.5 g. of material was dyed in 40 ml. of dye liquor at $80 \pm 1^\circ\text{C}$. until equilibrium was attained.

TABLE I
Chemical Constitution, Molecular Weights, and Melting Points of Quinonoid Dyes

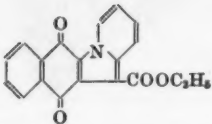
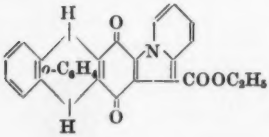
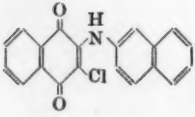
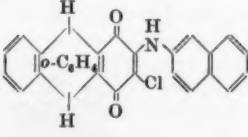
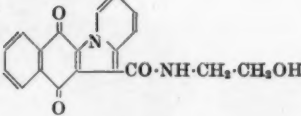
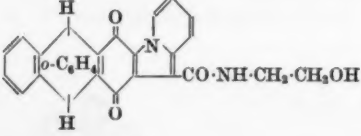
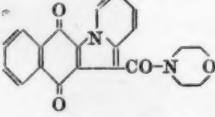
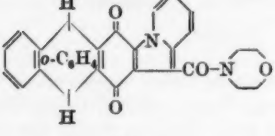
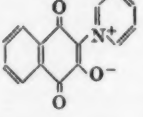
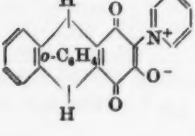
No.	Chemical Constitution	Method of Synthesis (Ref. No.)	Mol.Wt.	M.p. (uncorr.) (°c.)
SERIES I—DISPERSE DYES				
I		10, 11	319	154.5
II		—	445	262
III		12	333	199
IV		—	459	*
V		13	334	220
VI		—	460	266
VII		13	360	278
VIII		—	486	*
IX		14, 15	250	292
X		—	376	*

TABLE I—continued
Chemical Constitution

No.	Chemical Constitution	Method of Synthesis (Ref. No.)	Mol.Wt.	M.p. (uncorr.) (°C.)
SERIES II—VAT DYES				
XI		16	328	301
XII		—	454	*
XIII *		17	417	316
XIV		—	543	*
XV		18	467	292
XVI		—	593	*
XVII		19	758	*
XVIII		—	1010	276
XIX		19	624	*
XX		—	876	*

* These dyes do not melt up to 360°C.

After dyeing, the yarn was removed from the dye liquor, worked in a sintered-glass crucible (AG 3 × 4) with ice-cold distilled water, blotted between filter papers to remove excess water, dried over phosphorus pentoxide, and conditioned at 65% R.H. and $30 \pm 1^\circ\text{C}$. An accurately weighed quantity of the yarn (allowance being made for moisture) was dissolved in a known volume of dry acetone and the dye content determined with a Hilger "Spekker" photoelectric absorptiometer which had previously been calibrated with standard solutions of dye in dry acetone. Dye in the residual bath, together with that in the filtrate after washing, was also determined colorimetrically, after dilution with pure pyridine to give solutions in 25% aqueous pyridine.

Adsorption isotherms are illustrated in Fig. 5 for dyes I, III, V, VII, and IX, and in Fig. 6 for II, IV, VI, VIII, and X. The isotherms are linear, both with coplanar and non-coplanar dyes, up to saturation of the acetate rayon with the dye. Affinity values were calculated from the equation—

$$\Delta G^\circ = RT \ln \frac{[D]_f}{[D]_s}$$

where $[D]_f$ is the concentration of dye in the fibre and $[D]_s$ the concentration in the aqueous phase, expressed in moles per kg. of dry fibre and moles per litre respectively. This method of calculating the affinity of the dye for the fibre is not strictly correct, but in the absence of a definite value for the volume of internal phase in the fibre, and in order that the affinity values could be

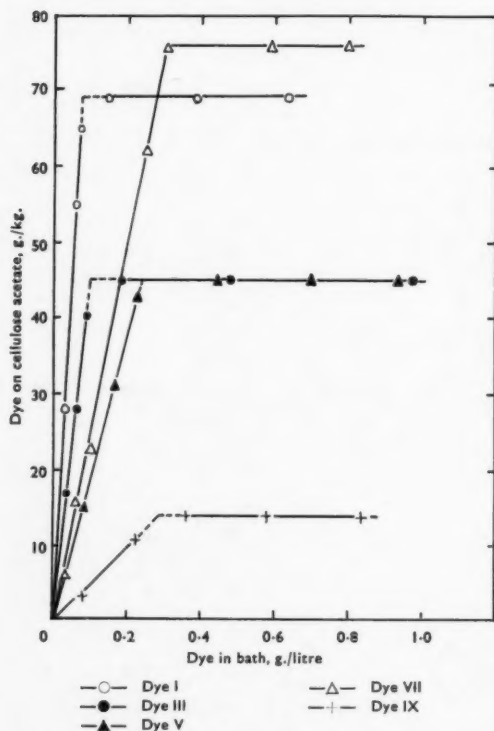


FIG. 5—Adsorption Isotherms for Coplanar Disperse Dyes

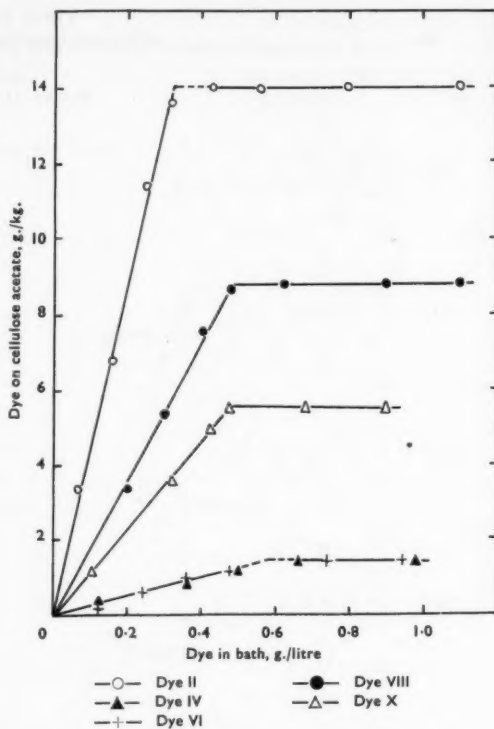


FIG. 6—Adsorption Isotherms for Non-coplanar Disperse Dyes

compared with those given by Bird and Harris²² for purified commercial disperse dyes, this method was employed in the present work.

With dyes XI–XX the method of dyeing and the calculation of affinity values were the same as those employed by Fowler, Michie, and Vickerstaff²³. Bleached cotton yarn was used, and the amount of sodium hydroxide and sodium hydrosulphite was adjusted so that the total sodium ion concentration in the final dyebath was 0.15 N. Adsorption isotherms obtained with XI, XIII, and XV are illustrated in Fig. 7, and the log-log plots of ionic concentration of the dye on the fibre and in the bath at equilibrium are given in Fig. 8. In the calculation of affinity, no correction was applied for the adsorption of hydroxyl ions by the cellulose, since only the dye concentration was varied, the amount of electrolyte being kept constant. The slopes of the linear log-log plots were close to unity, and the affinity values calculated over the range of dye concentrations studied were fairly constant.

Solubility of Disperse Dyes in Amyl Acetate at 80°C .

Crystalline dye in sufficient amount was added to 10 ml. of pure amyl acetate in a 100-ml. conical flask which was covered with a long-stem pear-shaped glass bulb. The flask was kept at $80 \pm 1^\circ\text{C}$. on a water bath for 8 hr., with constant stirring. The solution was then filtered through a sintered-glass crucible (AG 3 × 4) maintained at 80°C ., diluted with pure amyl acetate, and the concentration of dye determined colorimetrically.

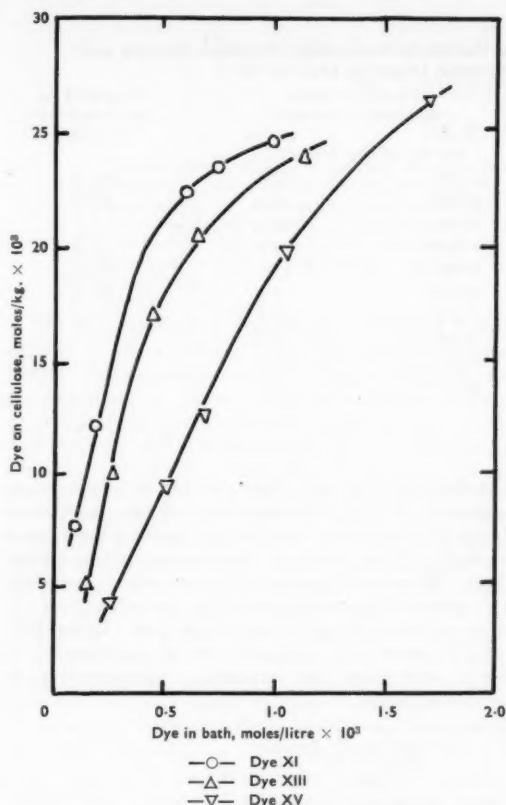


Fig. 7.—Adsorption Isotherms for Coplanar Vat Dyes

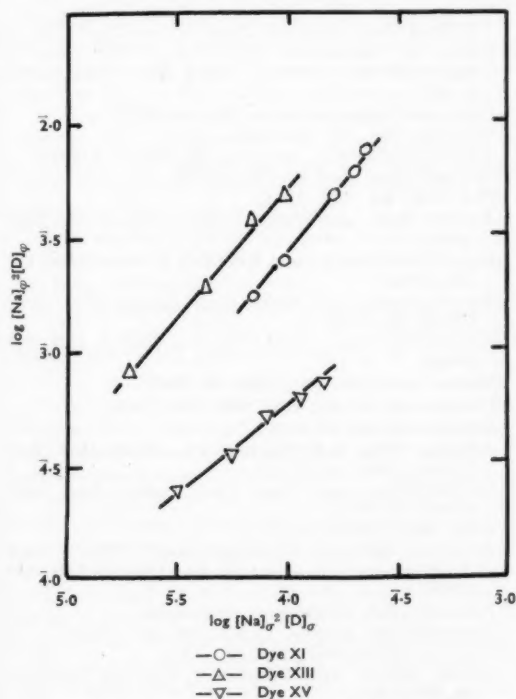


Fig. 8.—Adsorption of Coplanar Vat Dyes by Cellulose

RESULTS AND DISCUSSION

In Table II are given values for partition coefficient, affinity, saturation in secondary acetate, and solubility in amyl acetate, for dyes I–X at 80°C. In each pair the partition coefficient, affinity, and saturation value are considerably higher for the coplanar dye than for the corresponding non-coplanar dye. When hydrophilic groups, e.g. $-\text{CH}_2\text{CH}_2\text{OH}$ (V and VI), or ionic groups (IX and X), are present, both the affinity and the saturation value decrease, thus confirming the general view that the affinity of a compound for secondary cellulose acetate is decreased considerably when water-solubilising groups are incorporated in the molecule. Even with these pairs of dyes the effect of non-coplanarity in reducing substantivity is very marked. The affinity values of some of the coplanar dyes are comparable with those recorded by Bird and Harris²², but the saturation values are much higher than those of the aminoanthraquinone and aminoazo disperse dyes examined by Daruwalla and Limaye²¹. These coplanar naphthaquinone dyes should therefore be of technical interest for the dyeing of secondary acetate rayon, since they have good substantivity, as well as good building-up properties owing to their high saturation values.

It is generally considered that dyes for secondary cellulose acetate should possess a small molecule, very low aqueous solubility, hydrogen-bonding groups, and good solubility in specific organic solvents. However, examination of a large number of dyes has shown that these requirements hold good only in particular cases and are not of general application. Daruwalla and Limaye²¹ showed that, for an aminoanthraquinone or aminoazo disperse dye, there was no correlation between (a) chemical constitution, molecular size, or aqueous solubility, and (b) rate of transfer during steaming, saturation value, or partition between secondary acetate and a thickener. Bird²⁴ obtained a correlation between fibre saturation value and solubility in organic solvents, but only with solvents containing water, e.g. 20% aqueous diethylene glycol diacetate. In the present work solubility in amyl acetate has been found to have no bearing on fibre saturation value; even the non-coplanar dyes—which have low fibre saturation values—showed considerable solubility in this solvent. The average molecular dimensions of the disperse dyes in Table I are 12–13Å. in length and about 10Å. in width, so the dye molecules should be able to enter the water-swollen secondary acetate rayon without much hindrance. Consequently, the main factor responsible for the low partition coefficients and affinities of the non-coplanar compounds is probably their inability to assume the planar configuration which is essential if they are to lie close enough to the cellulose acetate chain-molecules for their secondary valency forces to become operative.

In the case of leuco vat dyes, although some of the work reported previously does indicate the importance of planarity in imparting affinity for cellulose, the examples cited are either not satisfactory, or a better alternative can be advanced to

TABLE II
Partition Ratios, Affinity Values and Saturation Values in Secondary Cellulose Acetate and Solubility in Amyl Acetate of Quinonoid Disperse Dyes at 80°C.

Dye No.	Partition Coefficient*	ΔG° (kcal./mole)	Saturation Value in Cellulose Acetate		Solubility in Amyl Acetate (g./litre)
			moles per kg. of dry acetate	grams	
I	998	4.85	0.218	69.4	35.0
II	39	2.57	0.0319	14.0	31.0
III	900	4.77	0.135	45.2	17.1
IV	2.4	0.61	0.0031	1.4	19.4
V	194	3.70	0.135	45.0	39.5
VI	2.6	0.67	0.0031	1.4	41.2
VII	216	3.77	0.212	76.2	—
VIII	17	2.00	0.018	8.8	—
IX	50	2.75	0.055	13.7	—
X	13	1.80	0.015	5.6	—

* Moles of dye per kg. of dry acetate
Moles of dye per litre of water

explain the changes in substantivity. Lukin's³ contention that 1,1'- and 1,2'-dianthraquinonylamine are substantive to cellulose because of coplanar configuration, whereas 2,2'-dimethyl-1,1'-dianthraquinonylamine is non-substantive because the *ortho* substituent makes the molecule non-coplanar, cannot be accepted entirely, since Courtauld molecular models show that none of these compounds is coplanar. A model of 1,1'-dianthraquinonylamine could only be constructed with great strain and considerable non-coplanarity; 1,2'-dianthraquinonylamine also is not planar. With 1-benzamido- and 1,4- and 1,5-dibenzamido-anthraquinones, substitution of methyl, methoxyl, ethoxy or chloro groups in the *ortho* position of the benzene ring has been found to hinder the dye molecule from attaining a coplanar configuration, thereby decreasing its affinity for cellulose⁴. However, when molecular models were prepared during this work, it was found that, in spite of the *ortho* substituents, the dye molecules could attain a planar configuration if the benzene ring containing the substituent was rotated by 180°. Attainment of coplanarity was impossible only when both the *ortho* positions were occupied by bulky substituents.

TABLE III
Affinity Values of Quinonoid Vat Dyes at 40°C.

Dye No.	ΔG° (kcal./mole)
XI	2.54
XII	<0.1
XIII	2.76
XIV	<0.1
XV	3.04
XVI	<0.1
XVII	3.9
XVIII	<0.1
XIX	4.13
XX	<0.1

In the present work, the pairs of vat dyes synthesised contain one coplanar and one non-coplanar dye, without other factors likely to diminish substantivity playing any part. The dye molecules are small enough to enter the water-swollen structure of cellulose. In the non-coplanar

compounds, the π -electron density is increased on account of the introduction of an additional benzene ring, and the total chromophoric area is higher than that of the corresponding planar dyes. However, in spite of factors which are likely to enhance their substantivity, the affinity of the non-coplanar dyes is extremely low (Table III), which proves the importance of coplanarity in leuco vat dyes for imparting substantivity to cellulosic fibres.

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The English Contribution to the Development of Copper-plate Printing

(The late) P. C. FLOU—

Copper-plate printing of textiles was first introduced by Francis Nixon at the Drumcondra works, near Dublin, in 1752, and by 1754 the secret had been transmitted to England. For the next twenty years it remained virtually a monopoly of the Dublin and London printers.

An examination is made of the difference between seventeenth century non-fast copper-plate printing of maps and handkerchiefs and the copper-plate printing with fast dyes by Nixon and his contemporaries. Examination of patents enables some description to be given of the development of textile copper-plate printing presses.

The introduction of copper-plate printing in the middle of the eighteenth century radically transformed the appearance and the status of European printed textiles. Prior to this, all printing on linen or cotton had been done with wood blocks, which had set very definite limits both to the delicacy of line that could be achieved and to the size of repeat which could conveniently be printed. The use of engraved copper plates immediately made it possible for the first time to obtain any desired degree of fineness in the drawing, and enlarged the maximum convenient size of repeat from less than a foot square to over a yard square. It is not surprising, therefore, that the new technique quickly led to the development of an entirely new type of printed fabric, decorated with elegant pictorial designs with figures, landscapes, and architecture, often incorporating mythological, romantic, theatrical or commemorative scenes of a type completely beyond the scope of the humbler wood-block printer. The extent to which these copper-plate designs differed from the wood-block prints is measured by the fact that they have always been treated by subsequent generations as forming an exclusive category, set apart from the routine printed cottons and chintzes, and referred to as *toiles*, and most frequently as *toiles de Jouy* after the leading French eighteenth century factory.

In view of this clear-cut demarcation of copper-plate textiles, both in appearance and esteem, it is surprising that almost nothing has been written about the precise time or place at which the new technique was first introduced, or about the various chemical and mechanical problems which its development involved, and that the little that has been written is often wrong. An extreme case is to be found in the article on "Textile Printing" in the current edition of the *Encyclopaedia Britannica* which gives the date of its invention as 1770, which, as we shall shortly see, is nearly twenty years too late; attributes the invention to Thomas Bell, who really invented roller printing; and states that "it has always been limited to handkerchiefs and designs made up of detached objects", which, as we shall also see, ignores the existence of many hundreds of the most interesting printed textiles. Even in France, despite the continued reputation of the *toiles de Jouy*, we get little guidance, and it is possible to search through Henri Clouzot's six different volumes¹ and innumerable articles about Jouy textiles without finding any statement as to who invented copper-plate textile printing and where, or even as to when the invention was first introduced at Jouy.

German writers are no more informative. Karl Reinking, who published admirable and detailed studies of the origins of wood-block printing and roller printing², never wrote a word on copper-plate printing, while Forrer, in his two standard works, completely confuses copper-plate printing and roller printing³.

In view of these uncertainties it is a relief to turn to the precise and clearly documented evidence assembled by Mrs. Ada Leask (Miss Ada Longfield), the Irish antiquary, whose researches are unfortunately too little known, and are mostly buried away in the publications of various learned societies in Ireland. In a paper published in 1937⁴ she established beyond question that in Ireland copper-plate printing on textiles was first introduced in 1752. The first evidence is an advertisement which appeared in *Faulkner's Journal* for 3rd October 1752 stating that "Drumcondra printed Linens, done from Metal Plates (a method never before practised) with all the Advantages of Light and Shade, in the strongest and most lasting colours" could be bought from George Gibbins at the Hen and Chickens in Werburgh Street, Dublin. Such an announcement would not, of course, provide positive evidence on its own, but it is very fortunately supported by a letter written two months later to her sister by Mrs. Delany, the well known diarist, stating that "Burke made me go with her to Drumcondra, half-a-mile off, to see a new manufactory that is set up there of printed linens done by copper-plate, they are excessively pretty". Moreover there is further confirmation from other independent sources. Thus the guard-books of the London Society of Arts contain a letter (communicated to me by Mrs. Leask) in which a senior member of the Dublin Society, writing to a London member on 13th June 1756, categorically states that copper-plate printing on textiles was invented by a certain Nixon, one of the proprietors of the Drumcondra printworks; and perhaps the most convincing testimony of all is that provided by Nixon's own competitors, several of whom, as quoted by Mrs. Leask, acknowledged him as the inventor.

By adding a few odd English references to the information already collected by Mrs. Leask in Ireland, we can piece together a tolerably clear picture of Nixon's career. Shortly before the copper-plate invention in 1752, he seems to have set up the Drumcondra printworks with a Theophilus Thompson, of whom we know almost nothing. In 1755 the enterprise was in full swing, judging by a petition sent to the Dublin Society by

the two proprietors, but by March 1757 it had closed down, as shown by advertisements announcing the sale of "the remaining Part of the Drumcondra printed Goods, consisting of Linens, Cottons and Lawns for Gowns, large Hangings, Chair-covers and Window-Curtains". The explanation for this sudden closure is certainly that given by Samuel Dixon, the proprietor of the competing Leixlip factory, in a petition to the Dublin Society in which he stated that the Drumcondra factory "grew into such immediate Repute that the Proprietors were prevailed on, by large Sums, to transfer the Manufacture to another Kingdom". It seems clear, however, that before himself moving over to England—presumably between 1755 and 1757—Nixon had prepared the way by imparting the copper-plate secret to an Englishman, for the author of the letter to the Society of Arts already quoted, states in 1756 that the new technique has been "transplanted to England by Mr. Amiand, who is admitted into the Secret by Nixon". This was George Amyand (the correct spelling), a wealthy Russia and East India merchant, who was also M.P. for Barnstaple⁵. He seems to have started a printworks at Phippsbridge, near Merton Abbey, Surrey, around 1752, and we can assume that Nixon joined him there after leaving Ireland, for in the earliest available London Directory (Mortimer's, 1763) the firm of "Nixon and Amyand" is listed as operating at Merton Abbey. Nixon himself died in 1765, aged 60, his tombstone bearing an inscription describing him as "the first that perfected copper-plate callicoe-printing"⁶. After his death the firm continued to operate as "Nixon & Co." although it is not quite clear under whose management, for Amyand, who had been created a Baronet in 1764, died in 1766, and John Anthony Rucker, another wealthy Russia merchant, who had been taken into partnership in 1765, seems to have concentrated mainly on his various City interests. The firm was finally sold up in 1789⁷.

When Mrs. Leask first elucidated the details of Nixon's Irish career, she did not feel justified in going so far as to claim that because he was the first to use copper plates in Ireland, he could equally be claimed as the inventor for the British Isles as a whole. It seems, however, that such a claim would be justified, for although there is an astonishing absence of any information about English calico printing in the obvious periodical literature of the mid-eighteenth century (apparently as a result of the printers' anxiety for their trade secrets), there is one unimpeachable later reference, namely a petition submitted to the House of Commons by the London printers in 1782, in which they categorically state that copper-plate printing was first introduced in 1754⁸. This coincides so neatly with the date at which Nixon must have transmitted his secret to Amyand, that we can take it as conclusive proof that no-one in England had forestalled Nixon's discovery.

The best evidence that the invention was successful is to be found, not in organised petitions, but in the ephemeral comments of satisfied

customers. The earliest so far traced dates from 1758, when Benjamin Franklin wrote from London to his wife in America, describing among other things bought for her "56 yards of cotton printed curiously from copper-plates, a new invention, to make bed and window curtains: and 7 yards of chair bottoms, printed in the same way, very neat"⁹. It is significant that this regard for copper-plate printing as "a new invention" continues well into the 1760's. For instance, in 1761, the entry under "Calico-printer" in Joseph Collyer's *Parent's and Guardian's Directory* includes the statement that "there has lately been a new improvement made in the art, by printing some fine cottons from copper-plates, which have a good effect", while Mortimer's already-quoted 1763 *London Directory* states, in the sectional heading for "Callicoe-printers" that "this art may be said to have arrived at the meridian of its perfection in England especially since the late improvements of printing cottons and linens from Copper Plate engravings instead of wood cuts".

The most convincing evidence on all these points is, of course, actual surviving textiles. At the time when Mrs. Leask first published her findings, no Irish copper-plate textiles had been traced. Since then she has, it is true, unearthed two interesting examples undoubtedly printed in Ireland¹⁰, but both dating from the 1780's and therefore considerably too late to have any connection with Nixon, or indeed with those other Dublin factories (Temple-Oge, 1758; Leixlip, 1758; Richardstown, 1765) which are known to have taken up copper-plate printing shortly after Nixon's invention. Definite evidence from England was hardly less meagre, and Donald King, in an article in this *Journal* in 1955¹¹, correctly summarised the then situation by describing surviving English copper-plate textiles as "rare". In that year, however, the position was transformed by the discovery, during the preparations for the exhibition organised by the Victoria and Albert Museum at the Manchester Cotton Board Design Centre, of many hundreds of paper impressions from early English textile-printers' copper plates, and since then of large numbers of actual textiles in American collections. Among this mass of new material is much that is directly relevant to Francis Nixon.

The first Nixon material to be found consisted of nearly one hundred impressions from Nixon's copper plates included in three large pattern-books in the collection of the Musée de l'Impression sur Étoffes, at Mulhouse, the centre of the Alsatian printing industry¹². In accordance with the normal eighteenth century practice, these patterns were printed on paper rather than on cloth as providing a cheaper and more convenient method of supplying customers with samples. The Mulhouse volumes appear to have been put together in the late eighteenth century by Alsatian printers who had collected samples of the work of their various English competitors. Of Nixon's copper-plate impressions, about forty were of large patterns suitable for furnishing fabrics, while the rest were of smaller dress patterns. Although the organisation of some of the designs

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FIG. 1—A textile in the Victoria and Albert Museum printed from a copper plate by Nixon & Co. at Phippsbridge, near Merton, Surrey, in about 1770. It corresponds with an impression marked "87" in a Nixon pattern-book in the possession of Mr. Ronald Baker. It had previously been regarded as the work of Oberkampf at Jouy



FIG. 2.—A textile in the Schuyler Mansion at Albany, New York State, which has always been attributed to the Drumcondra works, outside Dublin, at which copper-plate textile printing was invented in 1752. It may well be the earliest surviving copper-plate textile. (In the absence of the original, the photograph has been taken from the exact reproduction made by Scalamandrè Silks of New York)



FIG. 3—A textile in the National Museum of Ireland printed from a copper plate by John Collins at Woolmers, Hertfordshire, in 1765, as shown by the inscription on the base of the arch. The plate was later taken over by Nixon & Co., who printed it with the inscription erased, as shown by an impression in Mr. Ronald Baker's pattern-book, and a surviving textile in the Musée de l'Impression sur Étoffes at Mulhouse, Alsace



Fig. 4.—A paper impression from a copper plate included in Mr. Ronald Baker's Nixen & Co. pattern-book. It will be seen that the inscription "H. Roberts sculpt" above the keystone of the arch has been erased, thus indicating that the plate had been taken over, presumably from John Collins of Woodlams, for whom Roberts worked as an engraver.

is rather unsophisticated, it is certainly not primitive, and the standard of the actual engraving is very high.

In 1957 these impressions were supplemented by the discovery that the remarkable collection of Mr. Ronald Baker, the Crayford textile-printer, included a similar pattern-book consisting of twenty-two Nixon paper impressions for large furnishing designs. As eight of these duplicated those already found at Mulhouse, the total number of Nixon copper-plate designs now known is about one hundred and ten. The discovery of these impressions has already made it possible to identify nine surviving textiles, in England, America, and Sweden, as having been printed by Nixon, including one which has always previously been attributed to Oberkamp's factory at Jouy (see Fig. 1). There must certainly be others so far untraced.

Unfortunately there is no means of dating either these Nixon impressions or the actual textiles with any precision. Internal evidence shows that some at least cannot be earlier than 1765, and it seems probable that they all date from between about 1765 and 1775. There is, however, one textile in America which could perhaps be an example of Nixon's work at Drumcondra¹³. It bears a copper-plate design which, although not actually corresponding to any of the Nixon paper impressions, is extremely like them both in style and spirit (see Fig. 2). Moreover, as it has always been claimed as a Drumcondra print—no doubt on the basis of some family tradition—long before any information about these Nixon paper impressions had been published, there seems good reason to accept the attribution. If we do so, then we must regard it for the present as the earliest surviving example of copper-plate printing.

If these Nixon examples left us in any doubt about the ability of the Irish and English printers to produce fine copper-plate textiles in the mid-eighteenth century, it would be decisively dispelled by two exceptional textiles which conveniently incorporate their actual date of production in the design. These, bearing respectively the dates 1761 and 1769, were the work of Robert Jones's factory at Old Ford in Poplar¹⁴. They show an exceptionally high standard in the drawing, engraving, and printing, and with vertical repeats no less than six feet high they well demonstrate the revolutionary effect of the new invention on textile design. The 1761 design, of which more than twenty different examples have survived, already demonstrates such a mastery of the new technique that we must conclude that Jones had already discovered Nixon's secret several years earlier. Unfortunately, however, it has not so far proved possible to discover just when Jones started using copper plates—or indeed when he first started printing—so that we have no means of judging how quickly this followed either on Nixon's original invention in Ireland or on Amyand's transplantation of it to England.

Two other textiles, also incorporating dates in the 1760's, are particularly interesting as providing further evidence of Irish participation in early

copper-plate printing developments. These bear inscriptions showing them to have been printed by John Collins at Woolmers in Hertfordshire in 1765 and 1766¹⁵ (see Fig. 3). The Irish link arises from the fact that, as Mrs. Leask has pointed out, a John Collins was one of the proprietors of the Temple-Oge works in the Dublin suburbs, which is known to have printed from copper plates during its short existence from 1757 to 1759. In view of the fact that the impartial Dublin Society thought highly of its work in 1759, it seems likely that its precipitate closure was the result of success rather than of failure, and that its proprietors followed those of Drumcondra in moving over "to another Kingdom". Certainly the date fits in very well with the fact that John Collins first appears in Hertfordshire in 1761.

A curious link between Collins at Woolmers and Nixon at Merton, which would seem to strengthen the assumption of their common Dublin origins, is disclosed by some of the impressions in Mr. Ronald Baker's pattern-book. Although the book itself has "Nixon and Co." printed on the cover, it includes an impression of the 1765 Collins textile just referred to, but with the inscription carefully erased by cross-hatching¹⁶. Moreover, one of the other impressions clearly shows a similar erasure (see Fig. 4), and a third corresponds with a textile which, though uninscribed, has been attributed to Collins since the end of the last century. The evidence all indicates, therefore, that some at least of Collins's original copper plates were taken over by Nixon and Co., presumably when he went out of business in the late 1760's, and it seems reasonable to regard this as additional confirmation of the assumption that Collins of Woolmers and Collins of Temple-Oge were one and the same person.

We can now sum up the position in Ireland and England by saying that, in spite of many gaps in the picture, the combination of contemporary documents and of confirmatory surviving textiles makes it perfectly certain that Nixon was responsible in 1752 for an invention which introduced an entirely new type of printed textile. When we move over to the Continent we find far greater uncertainty and confusion. One would naturally expect some clear information concerning France, and in particular concerning Christophe-Philippe Oberkamp, the founder of the Jouy works at which the copper-plate technique received its most famous and elegant exploitation. In fact, however, recent French writers have contributed little to the subject, and the only definite statement remains that which is to be found in a biography of Oberkamp published as long ago as 1866¹⁷. This states categorically that the secrets of copper-plate printing were brought to Jouy in 1770 by Oberkamp's brother Frédéric, who had seen it in use at a printworks at Morat, near Neuchâtel in Switzerland. As the author apparently had access to Oberkamp family papers, there seems no reason to question this story, although no surviving Jouy copper-plate textiles can be positively dated earlier than 1783. In any case, no other writer has suggested a date earlier

than 1770, so that there can be no question of the Jouy works (which were not founded until 1759) having followed at all closely on the heels of Nixon and his contemporaries.

The information for other French and Alsatian printworks is hardly clearer than that for Jouy: 1774 has been suggested as the date for the first introduction of copper-plate printing at Orange, 1780 for Colmar, and 1782 for Mulhouse¹⁸, although in each case the evidence amounts to little more than a single isolated reference, unsupported by any clearly documented textiles. There seems no question, therefore, of France having anticipated the Irish discovery. Moreover, there seems to be confirmation of the British priority in the fact that the 1774 Orange reference is to an inventory-entry for "une machine anglaise à planche plate marchante". The same conclusion can perhaps be drawn from a remarkable French textile, the significance of which seems to have escaped all French writers. This is an excellently drawn and engraved copper-plate textile with a design of flowers and birds which has survived in two American collections. One piece, in the possession of Miss Elinor Merrell, bears the printer's mark of Gayet et Montgirod, of Sèvres¹⁹. Its special interest derives from the fact that, according to Clouzot, this factory only existed for eighteen months, during 1760–61²⁰, so that the textile can be regarded (if we exclude the uncertain Drumcondra piece) as perhaps the earliest precisely dated copper-plate example to have survived, with a possible lead of a few months over Robert Jones's 1st January 1761 design. Moreover this Gayet and Montgirod factory was apparently described as a "manufacture anglaise", which can presumably be taken to mean that it had the advantage of certain English affiliations not available to the numerous other factories springing up in France at that time. The probability that this advantage lay precisely in the secret of copper-plate printing seems confirmed by the fact that, despite its evident skill in the new technique, its secret never even leaked as far as the Jouy factory, less than four miles off, and seems to have been lost to France on the closure at Sèvres in 1761.

In view of the categorical statement about Frédéric Oberkampf's having learnt copper-plate printing at Morat in Switzerland in 1770, one would expect to find some positive evidence for its early use there. Nothing, however, is forthcoming. A fairly recent book on the whole history of Neuchâtel printing barely mentions Morat²¹, and the two other standard works on Swiss textile-printing do not hazard any statement on the earliest Swiss use of copper plates²². Nor do any documented Swiss eighteenth century copper-plate textiles seem to have survived.

The German facts are equally obscure. The tradition is that copper-plate printing on textiles was first introduced in about 1766 by Johann Heinrich von Schüle, of Augsburg, who is said to have gained an international reputation as a result. A full-length biography of Schüle, published in 1929, says, for example, that "he achieved a perfection with his copper-plate prints which

earned the astonished admiration even of the English"²³. Investigation shows, however, that these statements all go back to a single sentence in a naïve panegyric of Schüle written by a personal friend in the very last years of his very long life²⁴, and that even according to this account he was only able to take up copper-plate printing by importing two English printers and an actual copper-plate press from London at great expense. He can hardly, therefore, be counted as a pioneer of the technique, and this original statement, if accepted, merely confirms the fact that during the 1760's it was London that was the acknowledged centre of copper-plate printing. Moreover, not one single surviving copper-plate textile can be attributed to Schüle²⁵. As far as Holland is concerned there is equally little definite information.

It seems clear from this Continental evidence, therefore, that Nixon's use of copper plates in Dublin in 1752 really marks the beginning of the new technique, and that for over twenty years thereafter it remained virtually a monopoly of the Dublin and London printers. The most decisive confirmation of this is perhaps the statement of Jean Ryhiner, the Basle calico printer, in his 1766 notes, which form the earliest expert comments on the details of textile printing which have survived²⁶. In his remarks about English printing he particularly singles out copper-plate printing—with designs of landscapes and figures—for special mention, and clearly implies that this technique has not yet been copied elsewhere. In view of this acknowledged British priority it would be satisfactory if we could show why it was that such an important technological advance should have been initiated in backward Ireland. Unfortunately, however, the available evidence does not furnish an explanation. At first sight it seems tempting to find some connection between Nixon's discovery and the almost simultaneous discovery of copper-plate printing on ceramics, especially as this is usually attributed to John Brooks, an Irish mezzotint engraver²⁷. However, it seems impossible to sustain any actual connection between the two discoveries, and it must be admitted that the two processes, although both depending on the use of engraved metal plates, are rather dissimilar in that the ceramic procedure requires an intermediate paper-transfer. The only possible connection seems to rest on one single piece of evidence, namely a note, in an eighteenth century hand, on the back of a portrait in the British Museum of Robert Hancock, the leading engraver of copper-plate transfers for Battersea enamels and Worcester porcelain, which states that Hancock "engraved the first plate that was us'd for callico-printing"²⁸. Although Hancock's career has been closely studied by ceramic enthusiasts, no other clues link him in any way to textile printing, and it therefore seems best to regard the note as unsubstantiated. In the circumstances we can say nothing more definite than that the twin inventions—whether linked or not—both indicate that Dublin in the mid-eighteenth century was a much more lively centre for technological advance

that one might otherwise have supposed—a fact which can no doubt be partly placed to the credit of the enterprising Dublin Society, with its vigorous policy of encouraging national industries.

Having established Nixon's priority, we must now specify a little more precisely exactly what it was that he discovered, and in particular must make it clear that his invention was a technical achievement of some importance. Once more Ryhiner's notes provide valuable evidence. He specifically states that the actual mechanical printing process was perfectly straightforward, but that the key to the English secret lay in the "thickening" of the mordants to make it possible to print them from engraved copper plates. Although we do not have enough evidence to be certain as to exactly what thickeners were used, we can readily see that the appropriate thickeners to be added to the metallic mordants to render them sufficiently *fluid* to be sucked up from the engraved lines of a copper plate when the cloth was squeezed against it during the printing would have to be very different from the various gums traditionally added to the mordants to make them sufficiently *viscous* to be pressed against the cloth from relief-blocks without the danger of "bleeding". Ryhiner's notes make it clear that it was these new sort of thickeners for use with copper plates which Nixon must have developed.

Once this point has been clarified, it is possible to make a distinction, which we have so far deliberately avoided, between two quite different types of copper-plate textile-printing; namely, an early straightforward type already practised at the end of the seventeenth century, and the later chemically sophisticated type first introduced by Nixon in the mid-eighteenth century. Although no such distinction will be found in any of the standard histories of textile printing, it alone can explain certain surviving pre-Nixon textiles. Moreover, it is clearly adumbrated already in the earliest of all books on calico printing, namely the anonymous pamphlet entitled *Traité sur les Toiles Peintes* published in Paris in 1760²⁹. In this the author explains that copper-plate printing on textiles is quite simple, and can produce beautiful specimens such as maps finely printed on satin. He points out, however, that they are not fast to soap and water and should therefore be left out of account when commercial calico printing in fast dyes is being considered. This passage clearly refers to those pre-Nixon copper-plate prints which have survived from the late-seventeenth and early eighteenth centuries. The majority of these are, indeed, maps printed on silk, but academic certificates, religious broadsheets, and the like are also found³⁰. They were evidently intended to be framed like pictures, and are all printed in ordinary, unwashable, printers' ink. Into the same category fall one or two embroidery patterns printed on linen or cotton which have survived unused (no doubt many others have survived concealed beneath the stitches)³¹. As with modern embroidery transfers, there was obviously no need for these to be printed in fast dyes.

Only by drawing this sharp distinction between the early non-fast prints, and the later fast-dyed "toiles", can we explain a Dutch seventeenth century reference which is sometimes quoted as if it related to proper calico printing. This is a passage in a pamphlet published in Utrecht in 1690³², which describes how a certain Romeyn de Hooghe, an Amsterdam engraver, invented copper-plate printing on textiles in 1685, and had already made two million gulden profit. Although this statement is quite specific, it seems difficult to conclude from it that Holland had already discovered Nixon's secret in the seventeenth century, for were this so it seems inconceivable that there should not be one single piece of corroborative evidence in the form either of documents or of textiles to throw light on the development of this apparently profitable technique during the sixty-seven years which separated de Hooghe and Nixon. In fact the only conclusion must be that it referred to non-fast printing.

Two pieces of seventeenth century documentary evidence from England can be brought forward to support this interpretation, and incidentally to show that de Hooghe had no monopoly of copper-plate printing during the 1680's. The first is William Sherwin's patent of 1676, referred to in the first article. Although the fact seems hitherto to have escaped notice, the actual wording of the patent reads exactly like a specification for copper-plate printing. The patent was granted for "a new way of printing of broad calicoe and Scotch cloth with a double-necked rowling-*presse*"—a description which would have applied very well to the standard copper-plate engraver's press. This was sometimes called a flat-bed press, sometimes a star-wheel press—from the large winch which the printer operated with arms and legs—and sometimes a rolling-press, from the two rollers whose mangle-like pressure squeezed the paper or cloth against the copper plate. The epithet "double-necked" is admittedly rather obscure, but could easily have referred to the fact that in some versions the rollers turned in pairs of adjustable collars in order to allow variations in the pressure. In any case the specification cannot possibly have been intended to apply to wood-block printing, for which no press of any sort is required. Further support for the assumption that the patent referred to copper-plate printing is provided by the probability that William Sherwin the textile printer can be identified with William Sherwin, a well known copper-plate engraver³³.

The second English reference dates from 1688, and takes the form of an advertisement in the *London Gazette*. In this a Mrs. Barret announced as for sale "a large collection of copper-plates engraven with great variety of statues and other curious ornaments for hangings, curtains, etc."³⁴. We have, of course, no means of knowing for certain what these can have been, but the matter-of-fact wording makes it clear that some form of copper-plate printing on textiles was already by this date (only three years after de Hooghe's Dutch discovery) such an accepted fact in London,

that the normal references to "a recent improvement", "lately discovered", "a great novelty" and the like, were out of place. If this well-recognised seventeenth century commodity was in fact a fast-dyed plate-printed cotton, then the acclamation with which Nixon's invention was acclaimed in the 1750's is quite inexplicable. The only alternative is to conclude that it was some form of non-fast printed textile, whether silk or cotton, and that both Sherwin's patent and de Hooghe's invention referred to the same type of product.

Having made this distinction between these unimportant seventeenth century copper-plate textiles and the real mid-eighteenth century "toiles", we must conclude with a further distinction between these latter and an elaborated form of copper-plate printing which developed in the early nineteenth century. This again has been completely ignored by all English writers on the history of calico printing, but is fully attested by contemporary evidence. Whereas our earlier distinction was a chemical one, between non-fast and fast-dyed printing, this later distinction is a purely mechanical one, between the traditional copper-plate press such as must have been used by Nixon and his contemporaries, and a developed, more streamlined, press which was first introduced in England just after 1800.

Although there are many Continental references to this development, no one of them describes it in full, and one is therefore compelled to seek information from several different sources. The most helpful is the diagram of a copper-plate textile-printing press (see Fig. 5) included in the Atlas which accompanied Persoz's monumental treatise on all aspects of calico printing (1846)³⁵. This makes it clear that by then two substantial modifications had been introduced which differentiated the textile press from the traditional paper-printers' copper-plate press, which has remained virtually unchanged since the seventeenth century. The first is the "D"-roller; the second, the reverse-ratchet. Persoz's own text is of no help in elucidating the history of these two innovations, but other Continental writers supply the missing evidence.

The introduction of the "D"-roller was a simple but decisive improvement. Its function was to ease the inherent difficulties of using the copper-plate press to print not on single sheets, for which it had originally been devised, but on a continuous roll. These difficulties arose from the need to reconcile the alternate forwards and backwards movement of the copper plate on its flat bed through the mangle-like rollers, with the continuous movement forwards of the roll of cloth from repeat to repeat. The forward movement of the copper plate was simple enough; as it passed between the rollers its upper, printing, surface came in contact with the lower surface of the roll of cloth, which was stretched taut round the lower circumference of the upper roller. The difficulty was caused by the need to get the plate back through the rollers to its original position without winding back the cloth at the same time. The

problem was solved by the neat device of substituting for the lower roller a special roller with part of its circumference sliced off (hence the English name of "D"-roller, from the shape). On the forward passage the round part of this lower roller squeezed the plate up against the cloth for printing in the normal way; for the reverse passage the roller rested with its flattened surface uppermost, thus allowing space for the plate to be pushed back without touching the cloth.

The only writer who has drawn attention to the importance of the "D"-roller in the development of textile printing is General Poncelet, whose report to the French Government on the machinery exhibited at the Great Exhibition of 1851 is in effect a detailed survey of the whole history of machine-designing³⁶. He squarely places the credit for this innovation where it undoubtedly lies, with Robert Kirkwood, an Edinburgh copper-plate printer, who took out a patent (No. 2683) for a "D"-roller in 1803, and points out that later Continental patents, such as that of Rawle of Rouen in 1814, all stem from Kirkwood's original idea.

The second modification included in Persoz's illustration is the reverse-ratchet—a small refinement, but one which for the first time ensured that the copper plate automatically registered for the next repeat without individual adjustment of the cloth after each printing. The ratchet could be pre-set according to the size of the repeat, so that the cloth moved on just the right distance to avoid either gap or overlap. This was actually trickier than might be supposed, for a small reverse movement was required to allow for the unengraved margin between the edge of the copper plate—which sets the rollers and the cloth in motion—and the edge of the engraved area—which has to join on for the repeat.

The earliest evidence of the reverse-ratchet is to be found in a patent (No. 3639) taken out in 1813 by Matthew Bush, whose specification describes it as a "turnback, which is raised at each turn by the lifting piece or nog, and sets the work fair for the joining". The patent, however, which is most frequently recognised abroad was a very detailed consolidating patent of 1825 (No. 5311)—one of several taken out by Matthew Ferris, a leading Middlesex calico printer—which combined and refined all the details of both Kirkwood's and Bush's patents. Von Kurrer, for example, in his *History of Calico-Printing*³⁷—the first that was ever published (1840)—especially refers to Ferris's patent and implies that it had radically affected all copper-plate textile-printing.

Strangely enough, these early nineteenth century English mechanical innovations seem to have so impressed Continental observers that they came to exaggerate their importance very greatly, and consequently to underestimate the achievements of the eighteenth century copper-plate printers. The most extreme example of this is to be found in J. H. M. Poppe's otherwise most meticulous and detailed *History of Inventions* (1828-9)³⁸, in which he actually states that copper-plate printing on textiles is a recent invention,

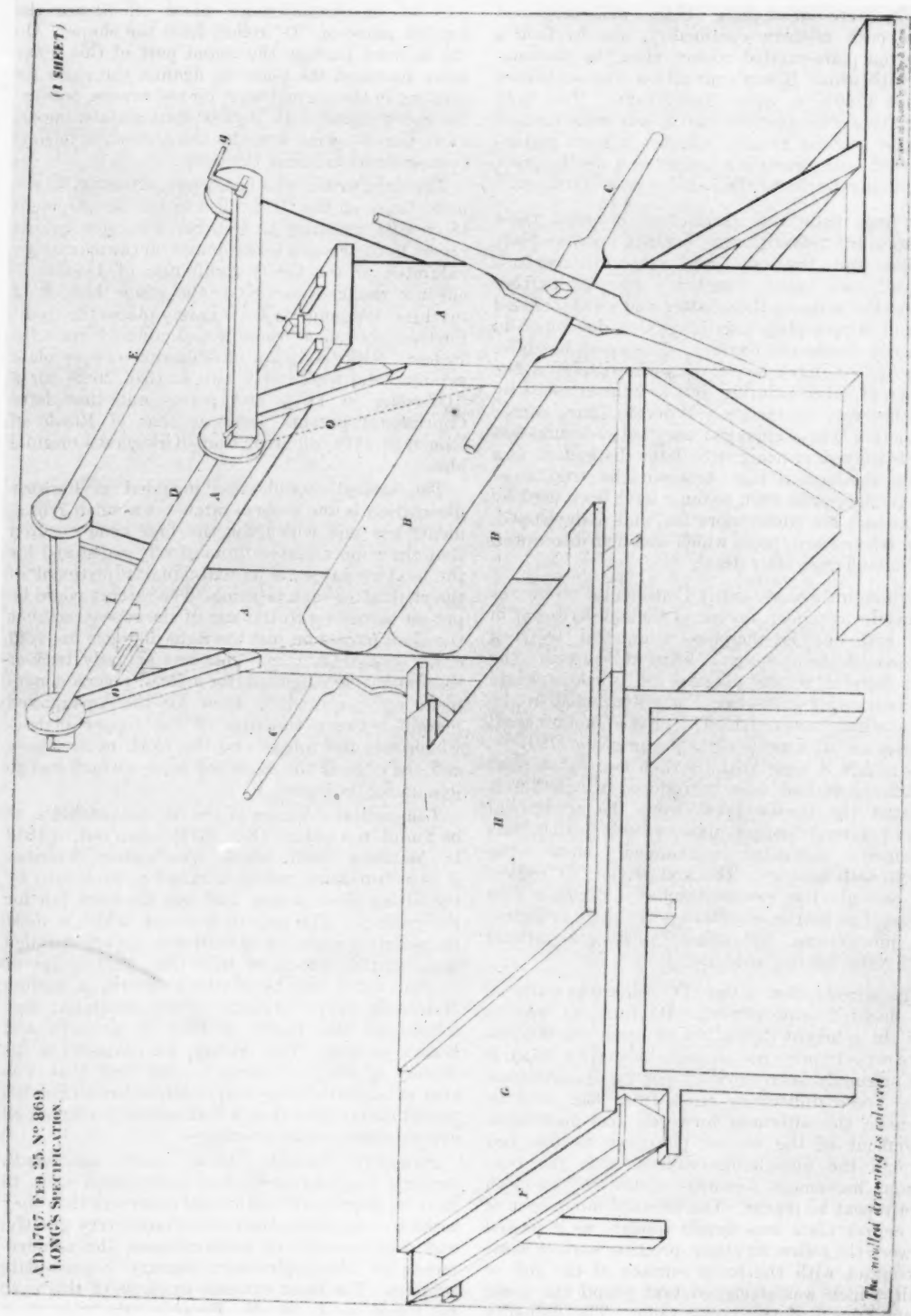


FIG. 6

first introduced by the English "about fifteen years ago"! A less extreme, but widespread, misconception was that prior to these English improvements the proper registration of repeats was so difficult with copper-plate printing that the technique had only been used for designs of detached sprigs and the like where an exact join was not required. This theory, which of course completely ignores the fact that the mid-eighteenth century English copper-plate designs invariably necessitated a carefully joined vertical repeat, is even found in the writings of experts such as Joseph Koechlin of Mulhouse (1829) and Persoz himself (1854)³⁹.

It is ironical that in reality it was precisely the despised primitive technique of the mid-eighteenth century which produced the finest artistic achievements in the whole history of textile printing, such as Robert Jones's prints from the 1760's, while the much-praised mechanical advances of the early nineteenth century accompanied the rapid decline and death of copper-plate textile-printing as an art. It would, of course, be naïve to suggest that the blossoming of the 1760's was the result of the primitive technique, or the later decline the result of the improvements. However, the two processes were undoubtedly linked, in the sense that the "golden age" of London copper-plate printing, between 1760 and 1785, reflected a leisurely atmosphere in which the best artistic results could be strived for, and finally attained, without a constant pressure for technical rationalisation as a means of speeding up production and reducing costs. With processes such as bleaching reckoned in terms of weeks, if not months, there must have seemed to be plenty of time to arrange the exact joining of repeats by manhandling the cloth into position after each impression, without any thought of "turnbacks", reverse-ratchets, and the like, while the plate could easily be carried back to its original position by hand, without the need for a "D"-roller. The primitive mechanics of these pre-industrial days can be judged from evidence such as the drawing of a copper-plate printing-press accompanying a minor patent taken out by Thomas Long of Mitcham (No. 869) in 1767 (see Fig. 6). As Long himself was a leading calico printer, we can take it that the drawing, though crude, gives a tolerably accurate picture of the sort of press in use by London printers such as Francis Nixon, Robert Jones, and John Collins. As will be seen, it includes no mechanical refinements whatsoever.

By the late 1780's the whole tempo of London production was being challenged. Incentives to speed up the printing were greatly increased by the saving of time resulting from the introduction of chemical bleaching, while at the same time the competition of Lancashire mass-production was becoming more intense each season. Many London printers went bankrupt. The survivors either attempted to lower costs by reducing their artistic standards, or turned to mechanical improvements to increase the output of the copper-plate presses. Both expedients failed. Once the standards of design and engraving were allowed to sink, copper-plate

prints could no longer maintain that special prestige which had previously kept up their price; even the most ingenious mechanical contrivances could never enable the intermittent motion of the copper plate to compete with the continuous motion of the new rotary machines. Devices such as those of Kirkwood, Bush, and Ferris no doubt postponed the final reckoning, and in the 1820's there seem even to have been partially successful attempts to drive copper-plate presses by power⁴⁰. In the long run, however, the cause was hopeless, and already by the 1830's we find references to copper-plate printing as "nearly obsolete"⁴¹. Thereafter it only survived for a few special jobs, mainly connected with handkerchief printing⁴². It can be counted as one of the earliest casualties of the industrial revolution, driven out of existence by the demands for faster and cheaper production.

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(Received 3rd November 1959)

Notes

- ¹ *La Toile Peinte* (Paris 1912), *La Manufacture de Jouy et la Toile Imprimée au XVIII^e Siècle* (Paris and Brussels 1926), *Histoire de la Manufacture de Jouy*, 2 vols. (Paris and Brussels 1928), *Les Plus Belles Toiles Imprimées de la Manufacture de Jouy* (Paris 1934), etc.
- ² On wood-block printing see "Die Entwicklung der Technik des Handdruckes", and on roller printing "Die Entstehung der Walzendruckmaschine" in *Melliand Textilber.*, 12, 462 (1931) and 9, 498 (1928) respectively.
- ³ *Die Zeugdrücke der byzantinischen, romanischen, gotischen und späteren Kunstepochen* (Strassburg 1894), and *Die Kunst des Zeugdrucks vom Mittelalter bis zur Empirezeit* (Strassburg 1898). The confusion is particularly evident on p. 71 of the latter work.
- ⁴ "History of the Irish Linen and Cotton Printing Industry in the 18th Century", in the *Journal of the Royal Society of Antiquaries of Ireland*, 67, Part II, 26-56 (1937).
- ⁵ A short biographical note on Amyand is to be found in *Kimber's Baronetage* (1771), Vol. III, p. 202.
- ⁶ Owen Manning and William Bray, *History and Antiquities of the County of Surrey* (London 1804), Vol. 1, p. 262.
- ⁷ See the advertisement of sale published by Mrs. Leask in the *Burlington Magazine*, 91, 71-3 (March 1949).
- ⁸ See the petition presented to the House of Commons by importers of cotton and calico printers in the London area protesting against the export of copper plates to India, *Journals*, 38, 893 (14th March 1782).
- ⁹ See *The Complete Works of Benjamin Franklin*, edited by John Bigelow (New York 1887), Vol. III, p. 7.
- ¹⁰ See "Edward Clarke, Linen-Printer, and the 'Volunteer Furniture'", and "Linen and Cotton Printing in the 18th Century at Ballsbridge, Dublin", in *Burlington Magazine*, 71, 179-180 (July 1942), and 79, 156-9 (June 1947).
- ¹¹ "Textile Printing in London and the Home Counties", *J.S.D.C.*, 71, 374-380 (1955).
- ¹² Owing to a mistake on the part of the author of this article, these impressions, when first discovered, were attributed to a mythical John Nixon, and were so described in the Catalogue of the Manchester Exhibition ("English Chintz: Two Centuries of Changing Taste", 1955).
- ¹³ Although the appearance of this textile is known from the reproduction of it made by the New York firm of Scalomandré Silks, it has so far proved impossible to trace the present whereabouts of the original.
- ¹⁴ For illustrations of these two Robert Jones textiles, see *J.S.D.C.*, 71, 376 (1955), Fig. 10 and 11.

- ¹⁵ For illustrations of John Collins textiles, see W. Gordon Hutton, *English Decorative Textiles* (1930), Plate 64, and *English Printed Textiles* (Victoria and Albert Museum, 1960), Plate 6.
- ¹⁶ Examples of this textile with the inscription intact are to be found in the National Museum of Ireland, Dublin, the Metropolitan Museum, New York, and several other American collections. A piece with the inscription erased is in the Musée de l'Impression sur Étoffes, Mulhouse.
- ¹⁷ Alfred Labouchère, *Oberkampf (1738-1815)* (Paris 1866), p. 35.
- ¹⁸ For Orange, see Henri Clouzot, *Histoire de la Manufacture de Jouy et de la Toile Imprimée en France* (1928), Vol. I, p. 134. For Mulhouse, see Charles Ballot, *L'Introduction du Machinisme dans l'Industrie Française* (Paris and Lille 1923), p. 291.
- ¹⁹ Miss Merrell's piece is illustrated in Clouzot, *op. cit.*¹ (1928), Vol. II, Plate 86. The other piece is at Colonial Williamsburg.
- ²⁰ Clouzot, *op. cit.*¹ (1928), Vol. I, p. 83-4.
- ²¹ Dorette Berthoud, *Les Indiennes Neuchâteloises* (Boudry 1951).
- ²² Adolf Jenny-Trümpp, "Handel und Industrie des Kanton Glarus" in *Jahrbuch des Historischen Vereins des Kantons Glarus*, 33 (1899) and 34 (1902), and Alice Dreyer, *Les Toiles Peintes en Pays Neuchâtelois* (Neuchâtel 1923).
- ²³ Jacques Waitzfelder, *Der Augsburger Johann Heinrich von Schüle* (Würzburg 1929), p. 18.
- ²⁴ Franz Eugen von Seida und Landensberg, *Johann Heinrich, Edler von Schüle* (Leipzig 1805).
- ²⁵ Forrer, *op. cit.*² (1898), illustrates some wood-block designs which he tentatively attributes to von Schüle, but without evidence. Erwin Bindewald and Karl Kasper *Fairy Fancy on Fabrics* (Brunswick 1951), illustrate and describe a page from a pattern-book attributed to von Schüle and kept at Künzelsau, his birthplace. On inspection the book proved to be of English origin, and to date from about 1820, that is, well after von Schüle's death.
- ²⁶ Long extracts from Ryhiner's notes were first published in Dollfus-Ausset, *Matériaux pour la Coloration des Étoffes* (Paris 1865), Vol. II. As his transcript is not always accurate I have checked the passage about English printing with an exact typescript of the original through the kindness of Monsieur P. R. Schwartz, of the Musée de l'Impression at Mulhouse, where the original manuscript is preserved.
- ²⁷ Cf. Cyril Cook, "John Brooks and his Engravings" in *Connoisseur* (May 1952).
- ²⁸ Cyril Cook, *The Life and Work of Robert Hancock* (1948), p. 13.
- ²⁹ Published as by M. Q***, who has been identified as the Chevalier de Querelles. A facsimile of the pamphlet was published by the Badische Anilin- und Soda-Fabrik, Ludwigshafen a. Rhein, 1925.
- ³⁰ An English example—a map dated 1686-8—was illustrated as Fig. 3, accompanying Donald King's article (J.S.D.C., 71, 376 (1955)).
- ³¹ A copper-plate embroidery pattern is illustrated as Plate 15 in *European Printed Textiles* (Victoria and Albert Museum, 1949).
- ³² *Der Neid und die Streitsucht nach dem Leben gezeichnet*, quoted by Jenny-Trümpp, *op. cit.*², p. 50.
- ³³ This suggestion was first made by Donald King. See Ada Leask, "Some 18th century Advertisements and the English Linen and Cotton Printing Industry" in *Burlington Magazine*, 91, 71-3 (March 1949).
- ³⁴ Quoted from B. Sprague Allen, *Tides in English Taste* (1937), Vol. I, p. 43. The fact that the author misinterprets the quotation as referring to varnished cloth as a substitute for window glass in no way affects the interest of the original quotation.
- ³⁵ J. Persoz, *Traité Théorique et Pratique de l'Impression des Tissus* (Paris 1846), Atlas, Plate VII.
- ³⁶ *L'Exposition Universelle de 1851; Travaux de la Commission Française*, Tome III, Première Partie, Première Section (1857), p. 183.
- ³⁷ W. H. von Kurrer and K. J. Kreutzberg, *Geschichte der Zeugdruckerei* (Nürnberg: 1st edition 1840, 2nd edition 1844).
- ³⁸ *Geschichte der Erfindungen*, 4 vols. (Dresden 1828-9), Vol. II, p. 4.
- ³⁹ Joseph Koechlin, *Bull. Soc. industr. Mulhouse*, 3, 256, (1829). Persoz, in his little-known report on the textile printing shown at the Great Exhibition of 1851, *Travaux de la Commission Française* (1854), Tome V, p. 9.
- ⁴⁰ See Report on Printing Machines by Jeremie Risler, *Bull. Soc. industr. Mulhouse*, 3, 250 (1829).
- ⁴¹ See article on "Calico-Printing" in the *Penny Cyclopaedia* of the Society for the Diffusion of Useful Knowledge (6, 150 (1836)).
- ⁴² It seems probable that the last use of copper plates for textile printing in this country was for printing the small number of commemorative silk handkerchiefs which were issued each year on the afternoon of the Derby, and bearing the name of the winner. Until 1946 these were printed from copper plates at the printworks of David Evans & Co. Ltd. at Crayford. Since then they have been screen-printed. The copper-plate press has unfortunately since been destroyed.

CORRESPONDENCE

The Publications Committee does not hold itself responsible for opinions expressed by correspondents

A Simple Test for Detecting Differences Among Colour Matchers

Colour matchers who are free from defective colour vision when tested by any of the recognised methods, nevertheless differ in their colour vision mainly because of differences in the amount of yellow macular pigment in the retinal tissue around the fovea and the degree of yellowing of the lens of the eye, the latter usually increasing with increasing age. These variations will cause arguments among colourists only when metameric matches are involved, and in such cases it is often very useful to be able to detect with certainty that differences in colour vision do exist and to demonstrate conclusively their existence to the disputants. An extremely simple method of doing this has been developed which requires only a G.E.C. Daylight Matching Lamp and a pair of blue patterns which,

to an average observer, are a metameric match in average daylight.

The G.E.C. Daylight Matching Lamp imitates average daylight by combining light from blue fluorescent tubes with the yellow light from tungsten filament lamps, the amount of yellow light being adjustable by means of a rheostat in the tungsten filament circuit.

When the pair of patterns is placed under the G.E.C. lamp, the dichroic pattern appears greener than the non-dichroic pattern when the amount of tungsten light is low, and redder when it is at the maximum. There is thus a rheostat setting where the dichroic pattern is neither redder nor greener, and this setting will depend on the macular pigmentation and the degree of yellowing of the lens in the eye of the observer, the greater the total

yellowing, the lower the setting required for a match.

Using the "floaters" and Pattern No. 7 of the blue Wain dichroic ladder¹ as the metameric match, this method has been found to be remarkably sensitive and reproducible, experienced colourists being able to repeat their settings to within four units on the rheostat scale, which is graduated from 0 to 100. The settings found for thirty-four colourists chosen at random varied from 50 in the case of a man aged 62 to over 100 for a man aged 29. To an average observer the appearance of the metameric pair when viewed at a rheostat setting of 50 was the same as when viewed under North light from a clear blue sky; at a setting of 100 they appeared the same as when viewed in direct sunlight.

This simple method should prove invaluable in colour matching laboratories where arguments among colourists could be due to differences in yellowness of vision.

K. McLAREN

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16th May 1960

¹ Jordinson, F., and Minshall, T., *J.S.D.C.*, **75**, 592 (Dec. 1959).

Variations in the Assessment of Light-fastness Exposures

We are pleased to note that McLaren, in his paper under the above heading¹, has obtained the

same result as we obtained with regard to variation in the assessments. The standard deviation of 0.53 found by McLaren agrees very closely with the standard deviation found by us².

It is incorrect to compare our standard deviation of 0.33 with McLaren's figure of 0.53. In our paper there are two components of variation, viz. the time factor, *T*, indicating variations between assessments of the same observer at different times; and the observer factor, *O*, indicating variation between observers. The sum of these factors represents a value comparable with McLaren's result. Thus—

Variation Components	Value	Corresponding Standard Deviation
<i>T</i>	0.17	0.41
<i>O</i>	0.15	0.33
<i>T + O</i>	0.32	0.57

It is the standard deviation 0.57 which should be compared with McLaren's value, 0.53. The standard deviation 0.33 merely represents the error between observers. Hence there is very good agreement between McLaren's result and ours.

U. BÜLOW
S. HORRDIN

BORÅS WÄFVERI AB.
BORÅS
SWEDEN
27th April 1960

¹ McLaren, K., *J.S.D.C.*, **75**, 597 (Dec. 1959).

² Bülow, U., and Horrdin, S., *ibid.*, **73**, 459 (Oct. 1957).

ERRATA

The Dyeing of Wool by Solvent-assisted Processes. W. Beal *et al.* (*J.S.D.C.*, **76**, 341 (June 1960))—In the last paragraph of the first column, for Table V read **Table IV**.

On p. 365 of the June 1960 issue the title of the pattern card under Sandoz A.G. should read **Drimarene-Y Reactive Dyes for Continuous Dyeing of Cellulosic Fibres** instead of Drimarene-Y Reactive Dyes for Prints on Cellulosic Fibres.

Notes

Reports of the Sections for 1959-60

HUDDERSFIELD SECTION

I have pleasure in presenting the Fortieth Annual Report of this Section.

Once again we have had a full programme, and all the lectures have been of a high standard. As in previous years, we have held joint meetings with the Huddersfield Textile Society, and with the Huddersfield Section of the Royal Institute of Chemistry. We also held a joint meeting with the Halifax Textile Society.

This year we had a visitor from Germany, Dr. J. Nüsslein, who renewed his acquaintance with us after a long lapse.

The average attendance at lectures has been approximately 35, but the Committee would like to see larger attendances, and if suggestions for

type of lectures could be made, they would be appreciated.

Our Annual Dinner, held 29th January, 1960, was again well attended, and we were all delighted to see our President, Mr. John Boulton, and our President-elect, Mr. F. M. Stevenson.

During the session we were asked to appoint an Honorary Treasurer, and Mr. Cyril Parker kindly consented to serve in this capacity.

I cannot close this report without thanking all who, by their help and advice, have made it possible to hold such a successful session.

HORACE TURNER
Honorary Secretary

LONDON SECTION

The London Section has had an eventful session. Nine lecture meetings have been held including the annual lecture at Luton and the Seventh London Lecture. Also included in this total are joint meetings with the Guild of Dyers and Cleaners and the Textile Institute.

The lectures in London have covered a wide field including such matters as the colouring of cosmetics and leather, and management in addition to textile subjects. We have again been privileged to hold our meetings at the Royal Society.

The Seventh London Lecture was given by Mr. E. A. Entwisle, director of the Wall Paper Manufacturers Ltd., on *Colour and Design in Wallpapers*. This was of the high standard associated with this series and attracted a large attendance.

A new feature introduced this session was short papers by Section members dealing with their own work and experience, and this was well received.

There was a good attendance at the lecture held at Luton but attendances at lecture meetings have generally been below the previous session. This state of affairs will be given careful consideration by next year's Committee.

The social events of the Annual Dinner and Dance and the Dinner after the London Lecture were well supported; attendances showing an increase on previous years. The Section was very pleased to have the company of the President and Mrs. Boulton on both these occasions. Excellent support has also been given to the Section by the Officers of the Society on several occasions and this is much appreciated by members.

Many members of the Section were pleased to participate in the successful Congress of the International Federation of Associations of Textile Chemists and Colourists held in London in September. The Fifteenth Mercer Lecture held in London was also well supported.

We have lost a number of members to the newly-formed West of England and South Wales Section and recruitment has not fully made up this loss, so that the Section shows a small decline in membership.

The session was concluded by the Annual General Meeting and Dinner.

H. W. TAYLOR
Honorary Secretary

MANCHESTER SECTION

Nine meetings of the Section have been held in Manchester this session, including one joint meeting with the Colour Group of the Physical Society. The attendance at these meetings has been of the order of 65 members with an extremely good response to the Ladies Evening meeting and the Printing Symposium. It must be stressed however that the attendance at the February meeting of the Section was the lowest on record for the past 16 years—22 members and guests.

Section meetings have been held in the Textile Institute and the College of Science and Technology and we are grateful to the authorities of these institutions for the accommodation and facilities placed at our disposal.

The Annual Dinner and Dance, held at the Grand Hotel, Manchester, on Saturday, 30th January 1960, was enjoyed by 296 members and guests.

The Knecht Memorial Prize for 1959-60 was presented at the February meeting of the Section

to Mr. K. Brocklehurst, who graduated B.Sc.Tech., 1st class Honours, at the College of Science and Technology, Manchester.

Five meetings of the Section Committee have been held during the past session. Mr. H. A. Turner has been re-elected as Chairman and Mr. A. S. Fern as Vice-chairman for the 1960-61 session. The Committee wish to record their sincere appreciation for the services of the retiring member, Mr. H. Hampson, and to welcome Mr. J. S. Ward to the Committee.

The membership of the Section has increased by 52 (including 24 juniors) during the past session to a total of 710 members.

It is with regret that we record the death of the following members of the Section—Mr. Mark Fletcher and Mr. Wilfred Kershaw.

K. MELDRUM
Honorary Secretary

MIDLANDS SECTION

During the last session lecture meetings were held at Nottingham, Loughborough, Leicester, Derby, Coventry, and Kidderminster. At our meetings we have had the pleasure of the company of members of the Nottingham Textile Society, Coventry Textile Society, British Association of Chemists, and Kidderminster Section of the Textile Institute. The average attendance has been almost 100 with more than 140 to hear Mr. Walter Beal at Loughborough and 120 for our half-day symposium on the dyeing of acrylics. Three of the lectures were given by Section members and a very wide range of subjects was covered.

The Committee has met five times during the year and the Honorary Secretary is very appreciative of the consistently high attendance and

enthusiastic support which he receives from all members and officers. For the second year a summer outing of the Committee members and their wives was organised and promises to become a popular annual affair.

The Annual Dinner and the Ladies Evening again received record support and were thoroughly enjoyable occasions.

Twelve members of the Section have been in attendance at various committees of the Society, some of them being members of several committees.

At the Annual General Meeting Mr. A. W. Carpenter resigned in accordance with Section bye-laws as Vice-chairman after being continuously in office in the Section for 17 years. The occasion was marked by the presentation of a silver fruit

bowl from those who had been associated with him on the Committee during this period. It is a pleasure to know that he still serves the Section as our only elected member of Council.

Mr. J. C. Wright was a popular choice as successor in the office of Vice-chairman. He has always been an enthusiastic member of the Section and we wish him every success during his next four years in office.

Members re-elected to the Section Committee were Messrs. D. O. Douglas, L. A. Telesz, G.

Whitehead, and D. Williamson. In addition we welcome to the Committee Mr. D. R. Graham and Dr. R. W. Richardson.

For the first time in 37 years a member of the Section, Mr. F. M. Stevenson, has been elected President of the Society. The Midlands Section is proud and pledges its support and best wishes for a successful and happy term of office.

J. RAYMENT

Honorary Secretary

NORTHERN IRELAND SECTION

The 1959-60 session just concluded has been very successful in the respect that we had a good series of seven lectures, which were much appreciated by members.

Our social functions were very well supported, and much enjoyed by members and their guests.

It is pleasing to report that all lectures were very well attended. We believe this was due to our choosing mainly lectures dealing with practical aspects of processing.

We shared two lectures with the local section of the Textile Institute, and we are pleased to record our excellent relationship with that body.

Our Annual Dinner Dance was held at Woodbourne House Hotel on 23rd October 1959. There was an attendance of more than 200 members and friends.

The Section Annual Dinner was held on 24th March 1960 at Grosvenor Rooms, Belfast, when we had an attendance of 84. We were very pleased to welcome the Honorary Secretary of the Society, Mr. L. M. Wood, Chairman of the Irish

Linen Guild, Mr. John M. Gray, and the Society's General Secretary, Mr. J. W. Nicholls. Musical items at this function were enjoyed by members and guests.

The Annual Golf Outing held at Hollywood Golf Club on 17th June 1959 was well attended, and enjoyed by members and their guests.

We were pleased to present to the Belfast College of Technology five guineas to be used for prizes for technical students attending Bleaching, Dyeing, and Finishing courses.

Our esteemed Chairman, Mr. W. Graham, died after a short illness on 27th February 1960. Mr. Graham was a very keen member of our Society, and was very proud of his position as Chairman. We miss him very much.

Mr. W. J. Watts was elected Chairman, and under his guidance and that of our enthusiastic committee we look forward to a further successful session.

JAMES PORTER

Honorary Secretary

SCOTTISH SECTION

The session which we have just concluded has been interesting and enjoyable. Eight lecture meetings were held in Glasgow and, in addition, we had our usual joint meeting with the local Section of the Textile Institute, again in Glasgow.

With two exceptions attendance has been somewhat disappointing compared with previous years.

During the session the Committee met three times.

Section membership shows a slight decrease over the year and although several new members were enrolled, we had a larger number of transfers, resignations, etc.

The Junior Essay Prize was awarded to Mr. W. A. Blue for his Essay *Towards Automation in Textile Colouring* and books to the value of three guineas have been presented.

Members were very disappointed that, due to extremely unfortunate circumstances, they were unable to meet the President; Mr. Boulton had arranged to occupy the chair at our January meeting and was flying from Manchester to Glasgow but due to adverse weather conditions, the plane was diverted to Edinburgh and he did not reach Glasgow until late evening.

The Annual Golf Outing was again successful, despite very unfavourable weather conditions.

In conclusion, appreciation must be expressed to our Chairman, Mr. J. G. B. McCallum, and all members of the Committee for their valuable assistance and support throughout the session.

F. ATACK

Honorary Secretary

WEST OF ENGLAND AND SOUTH WALES SECTION

The West of England and South Wales Section of the Society of Dyers and Colourists was inaugurated at the Grand Hotel, Bristol, on Friday, 26th June 1959. The President of the Society, Mr. John Boulton, took the chair, supported by the President-elect, Mr. F. Malcolm Stevenson, Officers of the London Section, and the General Secretary, and during the course of the meeting Officers of the new Section were elected.

It was subsequently agreed that the Committee should serve initially for two years, and shall be eligible for re-election except that the four members with the longest consecutive service shall not be eligible for election as Ordinary Members of the Committee until after the expiry of one year.

The elected Committee decided that, in view of the widely divergent trades embraced in the Section, it was necessary to provide papers with

as general an interest as possible, and attendances at the meetings during the session bore out this belief.

Of the attendance signing the register at meetings, in all 148 attended of whom 55 were attached to the textile trade, and it is felt that the Committee assessed the position fairly accurately

and that in future attention must be paid to other trades—particularly leather and paper.

In conclusion, any suggestions which ordinary members may have regarding papers for future presentation would be much appreciated.

J. N. LITTLE

Honorary Secretary

WEST RIDING SECTION

Under the Chairmanship of Mr. J. Rankin the Section has held nine lecture meetings and a One-day Conference on *Crease Retention and Crease Shedding* at The University, Leeds, on which occasion we were fortunate in having as speakers the two principal exponents of crease retention in wool fabrics, namely Professor J. B. Speakman of The University, Leeds, and Dr. A. J. Farnworth of the Commonwealth Scientific and Industrial Research Organisation of Australia. Mr. W. A. Bragg, of Warwick Chemical (Yorkshire) Ltd., and Dr. W. Lonie, of the British Rayon Research Association, who spoke on crease shedding, also made a useful and interesting contribution. The attendance was about 140. The average attendance at lectures, the topics of which were varied, was 63. Included in the lecture programme was the Geigy Bi-centenary lecture which was given by Dr. H. Ackermann. This was attended by the Officers of the Society and section members.

Our usual Supper and Smoker was not held this session, instead we held a Ladies Evening with a guest speaker, followed by a buffet supper. Our Annual Ladies Evening Dinner and Dance, whilst not quite as well attended, was once more a very enjoyable evening.

The entrance for *The Dyers and Finishers Association Competition* for West Riding Junior Members was again disappointing. Only two prizes have been awarded and these were presented at the Yorkshire sections' meeting held at the Bradford Institute of Technology. The winners were Mr. Roy Miles of Leeds University and Mr. Brian Nestor of Huddersfield.

A new member, Mr. Edwin Rowling, was welcomed to the Committee which has met on five occasions.

ESMÉE SMITH

Honorary Secretary

BRADFORD JUNIOR BRANCH

Six meetings were held at the Bradford Institute of Technology this session with an average attendance of 30 which was somewhat better than previous years.

A visit to Ciba Clayton's exhibition on *Reactive Dyes* held in Manchester proved of great interest to members.

The Annual Dance was held at Betty's Cafe, Bradford, on Friday, 4th December 1959, and a

good evening was had by all, but the absence of senior members was regrettable.

Members wish to record their sincere appreciation of the services of the retiring Chairman, Mr. R. S. Harding, the Honorary Secretary, Mrs. K. Cope, and Committee members, Messrs. B. Bennett, M. Dunsmore, E. Eastham, and H. Jaffri.

W. R. HERMES, JNR.

Honorary Secretary

LEEDS JUNIOR BRANCH

A programme of seven lectures and films was held during the session. The subjects covered a wide field and were well received.

We congratulate one of our members, Mr. R. Miles, who shared first prize in the *Dyers' and Finishers' Association Competition* with an essay on

The Impact of Reactive Dyes on the Dyeing Industry.

A highly successful 'smoker' was also held and has established itself as an annual event.

J. BUDDING

Honorary Secretary

MANCHESTER JUNIOR BRANCH

A very satisfactory session with an average attendance of 35 at the six meetings. Our sincere thanks are due to the following lecturers for providing most interesting and stimulating papers—Mr. F. M. Stevenson, Dr. A. S. C. Lawrence, Mr. G. L. Boardman, Dr. G. Landells, and Professor G. Porter.

The sixth meeting of the session consisted of short papers presented by members of the Branch,

and Mr. M. K. Smith was awarded the five guinea prize which was very kindly donated by the Manchester Section.

During the session formal bye-laws of the Branch were approved by Council, and there has been a marked increase in membership.

W. R. LEIGH

Honorary Secretary

SCOTTISH JUNIOR BRANCH

The Branch held six successful meetings this session at which the average attendance was 31. The Committee has noted that a large number of visitors attend these Branch meetings and it is encouraging to see that the lectures have stimulated the interest of textile workers who are not members of the Society.

Branch membership shows only a slight increase over last year and, at the Annual General Meeting of the Branch, members were asked to try and encourage young people in the dyeing industry in Scotland to join the Society.

The winner of this year's Scottish Section Annual Essay Competition was Mr. W. A. Blue with an

essay entitled *Towards Automation in the Dyeing Industry* and we should like to congratulate him on his success.

The Committee wishes to thank Mr. C. McNeil, our retiring Chairman, for his excellent leadership during his term of office and also the retiring members, Mr. I. Fleming and Mr. D. S. Hay, for their valuable services. We trust that, in the future, National Service commitments will no longer cause the premature resignation of our Junior Branch Committee members.

R. McDONALD
Honorary Secretary

Maurice Tordoff

Editor and Technical Officer of the Society



Maurice Tordoff, who takes up his duties as Editor and Technical Officer of the Society on 1st August 1960, was born in Bradford in 1931. After attending Bradford Grammar School he proceeded to St. Andrew's University where in 1952 he graduated with Honours in Chemistry. The next three years were spent in research at Leeds University where he was awarded his Ph.D. for a thesis on *Photosensitised Polymerisation of Acrylamides in Aqueous Solution*. He then became a Scientific Officer with the United Kingdom Atomic Energy Authority and later, in 1957, joined the Textile Institute as its Technical

Officer. During the last two years with the Institute he has been principally concerned with editorial matters, including the preparation of textbook MSS. for the printers. He was a Joint Staff Editor for Vol. 9 and 10 (1957 and 1958) of the *Review of Textile Progress* and since 1st January 1960 has also been Associate Editor of the *Journal of the Textile Institute* with particular responsibility for the Proceedings section. During 1959-60 he has been Honorary Secretary of the Manchester Federation of Scientific Societies.

Dr. Tordoff is married and has two children.

Annual Golf Outing of the Northern Ireland Section

This function took place on Saturday, 28th May 1960, at Castlerock Golf Club, Co. Londonderry. It was enjoyed by 37 members and friends under ideal conditions.

The occasion was memorable, being the first time the silver tray presented to the Section by the representatives of the various chemical manufacturers for annual competition amongst the members had been fought for.

The winner was J. Kershaw, J. Porter and J. Copeland being second and third respectively.

The visitors' prizes were won by J. Donaghy, C. Fulton, and G. N. Peters.

The Section Chairman, Mr. W. J. Watts, presented the prizes at a ceremony in the Club

House, and the Perpetual Trophy was duly christened in time honoured fashion.

Death of Members

We regret to report the death of Mr. L. W. Sidebottom and of Dr. C. M. Whittaker.

Meetings of Council and Committees June 1960

Council—1st

Finance and General Purposes—15th

Colour Index Editorial Board—20th

Publications—21st

Disperse Dyes—29th

COLOUR INDEX

Corrections

July 1960

VOLUME 1

Page				
1043	C.I. Acid Yellow 98	transfer to	Chrome Intra Yellow G ...	Vond
			C.I. Acid Yellow 130 (new entry)	
1151	C.I. Acid Red 134			
	under "The following brand is similar"	transfer to	Perlan Red 3B ...	Vond
			C.I. Acid Red 255 (new entry)	
1194	C.I. Acid Violet 7	transfer from	Acidine Red 6B ...	NCF
			"similar" to "the main list"	
1535	C.I. Mordant Green 12	transfer to	Calcochrome Green BB ...	CCC
			C.I. Mordant Green 11	
1589	C.I. Mordant Black 9	transfer to	Kayaku Chrome Black P2B ...	KYK
			C.I. Mordant Black 7	
1592	C.I. Mordant Black 15	transfer to	Java Chrome Acid Black L ...	Vond
			C.I. Mordant Black 80 (new entry)	
1714	C.I. Disperse Blue 3	transfer and from	Calcosyn Sapphire Blue 2GS ...	CCC
			Calcosyn Sapphire Blue R ...	CCC
			"similar" to "the main list"	
1737	C.I. Disperse Black 7	transfer to	Fenacet Diazo Black NS ...	G
			C.I. Disperse Black 1	

VOLUME 2

2024	C.I. Direct Yellow 50	transfer to	Triantine Fast Yellow RV ...	Vond
			C.I. Direct Yellow 91 (new entry)	
2034	C.I. Direct Yellow 77	for read	Garoeda Chrome Soga Yellow 26262 ...	Vond
			Garuda Chrome Soga Yellow 26262 ...	Vond
	C.I. Direct Yellow 78	for read	Garoeda Chrome Soga Yellow 26305 ...	Vond
			Garuda Chrome Soga Yellow 26305 ...	Vond
2048	C.I. Direct Orange 29	transfer to	Erie Fast Orange WS ...	NAC
			C.I. Direct Orange 102 (new entry)	
	under "The following brands are similar"	transfer to	Benzo Fast Orange WSA-CF ...	G
			Fenamin Orange AS ...	G
			C.I. Direct Orange 102 (new entry)	
2074	C.I. Direct Orange 99	for read	Garoeda Soga Orange 16262 ...	Vond
			Garuda Soga Orange 16262 ...	Vond
	C.I. Direct Orange 100	for read	Garoeda Soga Orange 16094 ...	Vond
			Garuda Soga Orange 16094 ...	Vond
2091	C.I. Direct Red 44	transfer to	Direct Bordeaux B ...	FDN
			C.I. Direct Red 13	

VOLUME 2—continued

Page					
2174	C.I. Direct Blue 12	under "The following brand is similar" transfer to	Trimaal Blue R Vond	
			C.I. Direct Blue 204 (new entry)		
2224	C.I. Direct Blue 151	transfer to	Kayaku Direct Copper Blue 2B	... KYK	
			C.I. Direct Blue 168		
2263	C.I. Direct Brown 1	for read	Naphtamine Brown 3G NCF	
			Naphtamine Brown D3G NCF	
2276	C.I. Direct Brown 33	transfer to	Direct Catechine B Vond	
			C.I. Direct Brown 197 (new entry)		
2283	C.I. Direct Brown 51	under "The following brand is similar" transfer to	Trifon Brown PGU Vond	
			C.I. Direct Brown 196 (new entry)		
2323	C.I. Direct Brown 182	for read	Garoda Soga Brown 16263 Vond	
			Garuda Soga Brown 16263 Vond	
	C.I. Direct Brown 183	for read	Garoda Soga Merah 16264 Vond	
			Garuda Soga Merah 16264 Vond	
2324	C.I. Direct Brown 187	for read	Garoda Chrome Soga Brown 26140 Vond	
			Garuda Chrome Soga Brown 26140 Vond	
	C.I. Direct Brown 188	for read	Garoda Chrome Soga Merah 26198 Vond	
			Garuda Chrome Soga Merah 26198 Vond	
2371	C.I. Sulphur Red 1	transfer to under	Kayaku Sulphur Bordeaux AR...	... KYK	
			C.I. Sulphur Red 3		
			"Applied by method 1"		
2389	C.I. Sulphur Green 11	transfer to	Sulfer Olive G Vond	
			C.I. Sulphur Brown 74 (new entry)		
2416	C.I. Sulphur Black 9	transfer to	Kayaku Sulphur Black BBX KYK	
			C.I. Sulphur Black 1		
			Page 2413 under "Applied by method 1"		
2533	C.I. Vat Brown 5	for read	Calcoloid Brown RRWP CCC	
			Calcoloid Brown RRP CCC	
2572	C.I. Azoic Diazo Component 2	for read	Fast Orange G Base SCW	
			Sanyo Fast Orange GC Base SCW	
2662	C.I. Azoic Blue 6	transfer to	Thiugen Blue BN SCW	
			C.I. Azoic Blue 8		
2855	delete C.I. Solvent Red 38	transfer dyes and all information to	C.I. Solvent Red 30		
2927	C.I. Developer 9	under "Constitution" for read	An N,N-Dialkyl arylamine		
			m-Diethylaminoacetanilide		

VOLUME 3

3349	under for read	42053	
		Discoverer—	National Aniline
		Discoverer—	Warner-Jenkinson Manufacturing Co., Ind. and Eng. Chem., 19, (1927), 497

VOLUME 4

4291	All Garoda products of Vond should be altered to Garuda in each item where there is a Part I number opposite.	for read	Garoda Chrome Soga Vond
			Garuda Chrome Soga Vond
		for read	Garoda Soga Vond
			Garuda Soga Vond

OBITUARY NOTICE

Leslie Gordon Lawrie

Leslie Gordon Lawrie was of that generation which served the Society so admirably between the two World Wars. He became a member of the Society in 1925 and took an active part in the affairs of the Manchester Section, serving on the Committee continuously from 1929 to 1942. In 1933 he was elected Vice-chairman of the Section and was Chairman during 1935 and 1936. L.G.L. will be best remembered as the joint author (with R. S. Horsfall) of the textbook *The Dyeing of Textile Fibres*, but he was also the author of *Textile Microscopy* and *A Bibliography of Dyeing and Textile Printing*. In addition he was a popular lecturer and the author of many papers to be found in the *Journal*. His work for British Dyestuffs

Corporation Ltd. and later I.C.I., Dyestuffs Division, was much concerned with the development of new products and new processes and his name was closely associated with the early problems of dyeing secondary cellulose acetate and later the development of the various types of auxiliary products. He retired from I.C.I. in 1953 and was happy to have the leisure to devote himself to his hobbies of fishing, book collecting, and gardening. Those of us who had the privilege of his friendship will remember the enthusiasm and energy he brought to bear on all matters in which he was interested; a charm of manner which could dispel the strongest opposition; and a nice appreciation of many branches of the arts.

H. BLACKSHAW

New Books and Publications

Methoden der Organischen Chemie
(Houben-Weyl)

Band V Teil 4

Halogenverbindungen
(Fortsetzung)

Edited by Eugen Müller. 4th edition 1960.
Pp. xlviii + 894. Stuttgart: Georg Thieme
Verlag. Price, DM 180.00.

The vast number of organo-halogeno compounds in existence today has necessitated their treatment in two volumes of which V/4 is mainly concerned with those containing bromine and iodine, V/3 being devoted to the fluoro- and chloro-analogues. Since halogeno-compounds are used extensively as intermediates for the preparation of more complex derivatives, a special section has been included to present the comparative reactivities of chlorine, bromine, and iodine compounds, with respect to their nucleophilic, electrophilic, and radical reactions, in vinylogous and phenylogous systems, and as reference for practical applications. Fluoro-reactivity is introduced where necessary. However a difference in treatment has been found expedient for V/3 and V/4, inasmuch as the former appeared to be more efficiently arranged according to reagents, whereas the data of the latter were better classified according to reaction types. Nomenclature is based as far as possible on the system adopted in Beilstein's classical work, and the literature has been searched up to and including 1958 in scientific and technological journals (over 1,200 consulted) and in the relevant patents.

The three main sections of the volume are: A₁. *Preparation of Bromo-compounds*, treated in subsections as follows: Survey of 16 brominating agents (with their general properties, preparation, purification, methods of drying, and application); Introduction of bromine by the element or by bromo-containing compounds (e.g. hydrogen bromide, hypobromous acid, alkali and organic hypobromites, iodine bromide, cyanogen bromide, phosphorus bromides, thionyl and acetyl bromides,

N-bromo-compounds, and organic bromo-complexes) as addition to unsaturated systems (with double or triple bonds); Entry of bromine by hydrogen exchange and also by displacement of other atoms or groups (e.g. of chlorine or hydroxyl); Preparation of bromo-compounds by synthetic and degradation reactions and by transformations of bromo-compounds with retention of at least one bromine atom.

A₂. *Preparation of Iodo-compounds*, dealt with in subsections as follows: A survey of iodination reagents as in the bromo-section; Introduction of iodine directly by the element, by iodine-containing compounds reacting with unsaturated systems, by exchange of hydrogen and of other atoms and groups, by special synthetic and degradation reactions, and by transformations in compounds containing polyvalent iodine (e.g. iodoso, iodo, iodonium) and of polyiodo compounds, with retention of at least one iodine atom.

A bibliography of 30 publications is given for A₂ and A₃.

B. *Reactivity and Reactions of Chloro-, Bromo- and Iodo-compounds*. As stated above, the treatment is comparative, and the name phenylogous is given to an activated system containing a halogen in *ortho*- or *para*-position to a nitro-, carboxyl-, cyano-, or sulphonyl-group. It is in two parts, viz. I *Reactivity*, dealing with the various mechanisms of substitution; and II *Reactions*, which consider elimination processes, and, in particular, those for hydrogen (by thermal cleavage and with reducing agents), hydrogen halide (by thermo-catalysis and basic reagents), and halogen exchange.

The author index contains 76 pages of names in three columns, while the subject index occupies 42 double-column sides. As with the previously published volumes, the fulsome appreciation already accorded to an earlier one (cf. J.S.D.C., 69 (1953) 133) applies with equal force to V/4, which, like the others, will have been eagerly anticipated

by the vast number of organic chemists who will now use it. They will find, in particular, that the various theoretical expositions which are included, wherever relevant, make the reading of such a factual volume extremely pleasant.

H. H. HODGSON

The Chemistry of Heterocyclic Compounds

Volume 14

Pyridine and its Derivatives

Part I

Edited by E. Klingsberg. London and New York: Interscience Publishers. 1960. Pp. x + 613. Price, for the four parts 367s. 0d. (subscribers 315s. 0d.).

The vast field of heterocyclic chemistry is still poorly documented compared, for example, with the now scarcely wider field of homocyclic aromatic compounds. The series of monographs on *The Chemistry of Heterocyclic Compounds*, under the general editorship of Arnold Weissberger, has already filled many gaps. An addition to this series, a four-volume treatment of *Pyridine and its Derivatives*, edited by Erwin Klingsberg, will be particularly welcome, since it deals with those heterocycles which have the greatest industrial importance and which command the widest range of interest. Part I, now available, comprises chapters on The Properties and Reactions of Pyridine and its Hydrogenated Derivatives by R. A. Barnes, and on Synthetic and Natural Sources of the Pyridine Ring by Frederick Brody and Philip R. Ruby.

In Chapter I (97 pp.) Professor Barnes deals adequately and readably with the structure and reactivity of pyridines, with the effect of the pyridine ring on substituent groups, and with partially hydrogenated pyridines. It is surprising, however, in a work intended to be comprehensive, to find physical properties despatched with such brevity. Absorption spectra, for example, have been granted only two pages and no illustrative spectra are recorded. There are 9 tables and 211 references.

Chapter II comprises sections on Pyridines from Natural Sources (50 pp.) and Pyridines by Synthetic Methods (392 pp.). This excellent monograph brings together for the first time a wealth of information of the greatest usefulness. The order and style of presentation are excellent. There are 132 tables, each summarising details concerning and literature references to all reported reactions of a certain type. The chapter has 1,851 references.

This work illustrates throughout its length all the more admirable features of current American practice in the writing of chemical monographs. The style is restrained, simple, direct, and readable. Occasional lapses from pedantic standards of grammar must be excused as a transatlantic prerogative. Formulae and reaction schemes are impeccably delineated, and are always adjacent to the corresponding text reference. Arabic formula numerals are used. The results of a literature search of formidable magnitude are presented concisely in tabular form, so that the finding of an original reference to a particular

reaction or compound is a simple matter. The text is free from errors and misprints.

This volume, with its three companion parts, will undoubtedly constitute the standard work on pyridine derivatives. It is a boon to chemists that this branch has at last been accorded comprehensive treatment; on the showing of Part I we will be doubly fortunate in being presented with a work that combines uniqueness with excellence.

D. G. LEWIS

Untersuchungen in der Leinengarnbleiche

By W. Rohs and I. Geurten. (*Forschungsberichte des Wirtschafts- und Verkehrsministeriums Nordrhein-Westfalen* No. 501.) Pp. 37. Cologne and Opladen: Westdeutscher Verlag. 1958. Price, DM 11.50.

The first part of the paper deals with the comparison of hank and package bleaching under similar chemical conditions. Bleaching was carried out under bulk conditions using 30s line and 14s and 20s tow yarns. The 30s line yarn bleached in hank form gave a higher viscosity and showed a little higher breaking load than the yarn bleached in package form. There were no significant differences with the 20s tow yarn; with the 14s yarn the package treatment gave a rather higher viscosity. The general conclusion is drawn that bleaching in package form gives a more uniform result and is safer chemically with coarser yarns. It is questionable, however, if the experimental data warrant this conclusion.

The second half of the paper is devoted to a study of the effect of the sequence of operations in bleaching 8s and 12s tow yarns in hank form. Variations in the following sequence were studied—calcium hypochlorite, sodium peroxide, and sodium chlorite. The results indicate that the greatest amount of chemical degradation is obtained when the yarn is bleached by a sequence in which treatment with calcium hypochlorite is the first operation. It is concluded that it is possible to bleach linen yarn to satisfactory viscosity and degree of whiteness provided the sequence is properly chosen.

D. A. DERRETT-SMITH

Vat Dyeing of Cotton Piece

Manchester: Imperial Chemical Industries Ltd., Dyestuffs Division. 2nd edition 1959. Pp. viii + 107 + 10 Plates. n.p.

This is a second edition of the monograph which appeared in 1947 under the title *The Application of Caledon Dyestuffs to Cotton Piece Goods in Open Width*. Compared with the earlier volume (61 pp.) it is considerably larger. This is not surprising, since room has had to be found for an account of acid-leuco padding and the Standfast Molten Metal dyeing process, as well as pad-steam methods, although this last is described only in outline since a full account has already appeared in the monograph *Continuous Dyeing by Pad-Steam Techniques* published in 1955.

The arrangement followed is radically different from that used in the earlier edition. Part I is concerned with the physical quality, dyeing

methods, dyeing rate, levelling properties and behaviour with dyebath assistants of the Caledon dyes and includes a comprehensive tabular summary of the properties and uses of the individual dyes. Part II covers cloth preparation and dyeing processes.

A large amount of data is presented in tables and there are a number of graphs and a few line diagrams. There are also ten plates showing mangles, jiggers, a washing and soaping range, and an 8-pass vertical slack drying machine. These are good of their kind, but would have been more useful if they had been accompanied by line diagrams.

Since this book is produced by a dye manufacturer, it is primarily concerned with that firm's products, but the mass of facts presented and the fundamental and up-to-date treatment of the whole subject of the application of vat dyes makes it a useful addition to the literature. Both practitioners and students of our technology have good reason to be grateful to dyemakers for publishing so many aids to its practice and study.

C. B. STEVENS

Färberische Eigenschaften von zur Herstellung von Verdickungen in der Stoffdruckerei bestimmten Stoffen

By P. Vogel. (*Forschungsberichte des Wirtschafts- und Verkehrsministeriums Nordrhein-Westfalen* No. 496.) Pp. 26. Cologne and Opladen: Westdeutscher Verlag. 1957. Price, DM 9.30.

This short thesis has to do with a problem that has for some time been considered as one of the potentially important factors which govern the transfer of dye from the impressed colour paste to the fibre-substance in textile printing with soluble colouring matters, particularly during drying and steaming of the print. As compared with dyeing, in which the competition for the dye is essentially between the fibre substance and water, in printing there is the possibility of distribution between fibre substance, thickening agent, and water.

In this work an attempt has been made to assess the "affinities" of two thickening agents for direct dyes. This is done by comparing the uptake of dye by viscose rayon, in conventional dyeing conditions, from water alone and then from water containing substantial amounts of dispersed thickening agent. (As a matter of interest, it should be noted that similar work was carried out by A. W. Doyle, M.Sc.Tech. Thesis, University of Manchester 1942, although the results were not published.) The significance of results reached in this way, in their application to the behaviour of printing systems, is somewhat doubtful, since this approach ignores to a great extent the very profound changes in dye concentration and in the structure of the starch dispersion during drying-down and steaming of the printed fabric. This seems to be a much more fundamental objection to the procedure chosen than the one which the author makes to the use of the convenient films of fibre-substance which have enabled dye partition to be determined under more realistic conditions than the present. It would, of course, have been a great achievement if the author

had succeeded in measuring transfer of dye to actual textile fibres while still maintaining the concentrations and other conditions which obtain in an actual print.

On other grounds than this, the report is a disappointing one, and should not have been made available to the general public. It has been presented as part of the requirements for the degree of doctor, and, as such, is no credit either to Herr Vogel or to the Institution in which the work was carried out. It would fall far short of the requirements for the M.Sc. degree in an experimental subject in an English university. There is the usual review of the relevant literature and a display of the wider circumstances and principles of textile printing which fills half of the thesis.

Much of the experimental work, although it may have been carried out with due care, seems to be badly misconceived. For example, in the dyeing experiments in which thickener is also present, the system seems to contain 0.1 g. of viscose, 30 g. of thickener and 1.5 l. of water. How results with these proportions can have any significance and indeed, how the dye associated with the thickener can be determined by a difference method with any real accuracy is difficult to understand. Unless the description lacks important details, the method of removing dye from the viscose with aqueous pyridine for estimation seems grossly over-severe. It is done in a Soxhlet-like apparatus at 100–110°C. and it is stated that no destruction of the dye takes place, but one wonders whether the solvent itself may not become slightly coloured in the process. The isotherm for absorption of dye by starch is said to be linear, and it is substantially so at 90°C. for a fairly wide range of equilibrium dye concentrations. At 60°C. with potato starch, there is, as might be anticipated, a great deal of scatter of the experimental points for the higher dye concentrations.

Many positive lessons are learned from the doings of foreign scientific institutions: for once, we are provided with a negative one, viz. that our custom of putting out accounts of academic research only in the form of a prepared submission to a learned society, where they must run the gauntlet of an independent system of refereeing, avoids many risks to the reputation of author and institution.

H. A. TURNER

Der Rakeltiefdruck

By A. Strakaty. Pp. 116 + 1 colour plate. 10th (Jubilee) edition 1959. Mauthausen an der Donau: A. Strakaty Druck und Verlag. n.p.

For many years, the mechanical aspects of the printing of paper and of textiles continuously by machines have shown many similarities. The deeper intaglio required for the more open structure of textiles led to the open scale for controlling the paste in the engraving together with the stipple for shading. The closer, smoother texture of paper, in which penetration must usually be discouraged, was adapted to lithography and the small shallow depressions produced by the screen in photo-mechanical work. However, in recent years, the

two industries have come much closer together. Great improvements in the properties of inks have rendered continuous colour printing on paper from rollers increasingly popular, and the industry has borrowed the doctor as a clearing agent from intaglio rollers. The textile printing industry has become increasingly interested in photoengraving, partly to overcome certain difficulties presented by the design, but much more because the speed with which screens could be prepared called for some answer from the engraver. Now we have the Star process in which the actual printing for the textile is first done on paper, and so there is a prospect that the very versatile resources of rotary photo-gravure will be called upon more and more in the future.

The present book is a straightforward factual outline of the various methods available for preparing the rollers and for printing in rotary photogravure by most of the accepted methods. Accompanying it is a review of much of the modern plant commercially available, mostly from central European sources, for various applications and with various performances. To a non-expert in the field it seems to be a fair, sensible, and able book. It may already be known to the textile machinery engineer. If it is not, he should consult it for it will contain much to interest and impress him. The printer also might find that a greater study of the field could provide him with the inspiration to devise new methods for some of his old problems.

H. A. TURNER

Handbuch für Textilingenieure und Textilpraktiker

Teildruck T 61

Grundlagen der Textilveredlung

By M. Peter. Pp. xi + 413 (including 58 advertisement pages). 8th revised and enlarged edition 1960. Wuppertal-Elberfeld, Germany: Dr. Spohr-Verlag. Price, DM 10.80.

This highly compressed small handbook contains, within the limits permitted by its format and size, a surprisingly large amount of information. Assuming that he could read German—and as an embryo technologist he should—it would be an admirable companion for the student of textile technology in the technical college and university. The author claims that he has attempted to deal with the fundamentals of most accepted procedures in textile processing, along with the appropriate plant and chemical auxiliaries.

With the term *fundamental* one might disagree, since profound theoretical treatments are really outside the ambit of what is essentially a practical handbook, but in its outlines of processes it is remarkably thorough. Also, although an explicit appraisal of the relative merits of competing procedures has mostly to give way to descriptions of the processes themselves, a fairly sound implied criticism is furnished by selection and emphasis. In all this the chief merit of the book is the fact that it does really attempt to be up-to-date, and mention is given to most of the topics that are developing and being discussed at the present time.

There are, for example, drawings or illustrations of new developments in machinery which do not seem to have appeared in a single textbook before. To take examples at random, there is the Henriksen circular carrier for dyeing hank yarn in package machines, the Obermaier Turbostat, the Bow ager, the Dornbuscher universal calender for coloured embossing, the B.D.A. confining-passage shrinking machine, the Artos stenter, etc. Most of this machinery is, understandably, from German sources (this may in part be connected with the large number of makers' advertisements interleaved in the pages of the appendixes), but it would improve the book if more examples from the U.S.A. and from other European countries were given. The finishing side of the subject receives much attention, and most of the modern finishing methods are mentioned although generally in catalogue form only. In appendixes, lists are given of the principal chemical and machinery manufacturers of the world whose products contribute to textile processing, and there is a full textbook bibliography.

H. A. TURNER

Removing Spots and Stains

By I. Mellan and E. Mellan. Pp. 95. New York: The Chemical Publishing Co. Inc. 1959. Price, \$4.50.

This book is of little value to the professional dry cleaner. At the most it could find a place on the home bookshelf alongside the "Handyman" and similar volumes of a "Do It Yourself" character. The most valuable advice is given in the introduction but the glossary of fibres in Chapter 2 is sadly incomplete. It comes rather as a surprise to find viscose rayon under the trade name of "Celanese" and no mention is made of the fact that Dacron is synonymous with Terylene. Cellulose triacetate does not get a mention. This fibre is very susceptible to certain chlorinated solvents and extreme care must be taken during spotting. Following the development of new fibres and finishes, spotting is rapidly developing into a science, with a result that careful and prolonged training of personnel is essential. The housewife cannot hope to accomplish, in the heat of the moment, successful stain removal using the advice in this book.

G. P. PEARSON

Chemical Analysis of Resin-based Coating Materials

Edited by C. P. A. Kappelmeier. London and New York: Interscience Publishers. 1959. Pp. xxvii + 630. Price, 147s. 0d. (\$19.50).

The task of the analyst in this field has never been easy: coating formulations have a strong, traditional connection with witches' sabbaths, and many ingredients are of complex constitution. The post-war years have seen a proliferation in coating materials and, though this has been paralleled by an increase in the analytical tools and methods available, there has been a growing need for a book such as this to draw together the threads. It is

sad that it must, so near to its publication, serve as a memorial to its devoted Editor.

There are three broad divisions in the book: the first two deal with the analysis of oil-based materials, and of lacquers, respectively; the third gathers together various chapters dealing with particular materials or analytical methods. The 21 subdivisions and their numerous subsections have been contributed by a number of distinguished workers, including the Editor himself. The materials considered range from starch, gums, rosin and the like, to polyesters, acrylics, and polyvinyls. Amongst the omissions are: soluble nylon, polyethylene and polypropylene, and polytetrafluoroethylene, but the emphasis in this book is upon paints and varnishes even though many of the materials considered are by no means confined to these uses. The analysis of solvents, pigments, plasticisers, and minor constituents such as wetting agents is also well covered. Analytical methods range, likewise, from old favourites such as the Prussian Blue test, Liebermann-Storch coloration, and dry ignition, to filter paper and gas-liquid chromatography, infrared and ultraviolet spectroscopy, polarography, and compleximetric titration. There are several tables, including systems for separation and identification on "group analytical" lines.

It is generally assumed that the coating materials are to be analysed in their initial form; the book would have benefited greatly from the inclusion of one section at least, dealing with the often difficult problem of separating the material from a substrate such as wood, metal, or a textile fabric. Many of us are all too familiar with such requests as "Let us know the composition of the coating, quantitatively too, and please return to us any material you have left over"—usually accompanying a square-inch sample of a coated material. To such sorely-tried mortals as these, amongst others, the detailed text and liberal bibliographies of this well-produced book will, despite the above small criticism, be as welcome as the brandy flask hanging from the St. Bernard's neck—neither do they need to contract frostbite to get it.

J. W. DUARTE

The Analysis of Fatty Acids and Fatty Alcohols Technical Publication No. 6

New Ferry, near Birkenhead: Price's (Bromborough) Ltd. Pp. 149. March 1960. Gratis.

This publication brings together in one volume methods of analysis which are used by one of Europe's leading fatty-acid producers in the examination of fatty acids and alcohols.

The first section contains a short introduction on the nature of fatty acids and fatty alcohols followed, in each case, by a comprehensive discussion of the analytical procedures adopted. A chapter entitled *Sampling Techniques* emphasises the need for representative sampling, particularly in the case of fatty materials where fluctuations in temperature can cause partial liquefaction or crystallisation. The second half of the book is given over to *Detailed Test Procedures*.

The analysis of fatty acids and alcohols is considered from the point of view of tests providing information on composition, quality, and behaviour in use. Most commercial fatty acids and alcohols are mixtures, consequently tests providing information on their composition are perhaps those most often carried out; certainly this section of the publication is the largest. Conventional tests involving observation of liquid/solid transitions, the determination of acid, saponification, iodine, and hydroxyl values, and mean molecular weights are fully described and their value and limitations are discussed. Although the above-mentioned characteristics throw considerable light on the composition of a sample and, indeed, often serve as a basis for specification and quality control, they do not give much idea of the structure and distribution of the chemical entities: it is pleasing, therefore, to find an adequate discussion of the value of ultraviolet spectroscopy and gas/liquid chromatography in the analysis of fatty materials.

Tests of quality are mainly concerned with the colour and purity of the sample. The determination of colour is a notoriously difficult subject which it is felt would ideally be carried out spectrophotometrically. For general purposes, however, the method used in Price's Laboratories is the Lovibond system, but other systems favoured by customers are included. Other tests giving a measure of the cleanliness and degree of refinement of the sample include determination of water, dirt, ash, trace metals, and unsaponifiable matter.

As tests for behaviour in use are dependent on the use to which the material will be put, this section is confined to the most common tests such as the Mackey test for oleines, the heat stability, and the colour of derived products.

Price's have produced a very readable and comprehensive account of their analytical methods. The lay-out of the experimental section in particular is extremely clear. A minor criticism is that the binding does not appear likely to stand up to the hard usage the book deserves. All users of fatty acids and alcohols should find this publication of great value.

A. H. MILBURN

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes.
Any publication abstracted may be referred to by members of the Society on application to
Dr. C. B. Stevens, Dyeing Department, Leeds University

Ciba Ltd.

CIBACRON YELLOW G—This reactive dye, described in this supplement to Pattern Card No. 3050/59, 'Cibacron and Cibacrolan Dyes on Wool', gives bright yellows, intermediate in hue between Cibacron Brilliant Yellow 3G and Cibacron Yellow R. It is recommended for use, alone and in mixtures, for bright yellows, greens, oranges, and scarlets. Fastness figures include—Light 6, washing (b) 3-4, heavy milling 3-4 (staining of cotton), potting 3-4 (staining of wool).

Durand & Huguenin S.A.

PANDURAN BLACK GR—This is a new type of mordant dye specifically intended for deep blacks on cellulosic materials and is especially recommended for fine outlines which are to be overprinted with solubilised vat dyes on stabilised azoic combinations. Chromium acetate is recommended as the mordant on cotton and Universal Mordant RC when printing viscose rayon. Full depth prints should be soaped at the boil. Any thickener suitable for chrome colours may be used and also modified petroleum emulsion thickeners and it may be fixed by either neutral or acid steaming. Contrary to the usual method of preparing chrome printing colours, Panduran Black GR must be mixed directly with the mordant and then hot water and urea added, the temperature throughout not exceeding 70°C. Copper vessels should be avoided as far as possible. It is not stable to reducing agents and not recommended for greys. Fastness figures include—Light 6, washing (c) 4-5, dry chemicking 4-5.

[Farbenfabriken Bayer A.G.]

ASTRAZON VIOLET F3RL—This cationic dye gives bright violets of very good fastness to light and wet treatments on acrylic fibres and builds up well to give deep dyeings. It is suitable for dyeing at 105-108°C. Levagol PAN is recommended as a restraining agent when dyeing pale colours. To ensure complete solution of the dye in the dyebath, it is recommended that the dye powder should be pasted up in a dry vessel with an equal amount of glacial acetic acid, and then diluted with a little hot water and the solution strained into the bulk of the water. Fastness figures on Dralon include—Light 6, washing (b) (140°F.) 4-5, milling (b) (severe milling) 5.

PERMAFIX BRILLIANT BLUE 3GL: PERMAFIX YELLOW 4G—These are the first two of a range of reactive dyes intended for dyeing and printing cellulosic materials. Yellow 4G gives brilliant golden-yellows and Brilliant Blue 3GL bright turquoise blues. Pad-dyeing liquors and printing pastes are set with urea, sodium alginate, and caustic soda and fixation is brought about by neutral steaming for 5-8 min. or dry heat at 140-150°C. for 4-5 min. Thorough rinsing and soaping at the boil is essential to remove uncombined dye but this presents no difficulty owing to the very low substantivity of these dyes. Solutions of these dyes are prepared by mixing the dye powder with the requisite quantity of urea and then either adding water at 20-30°C., followed by caustic soda solution, or by adding boiling water, boiling up as necessary, cooling to below 50°C. and then adding the caustic soda. In printing, white and coloured discharges and resists can be obtained. Leucotrope W improves the whiteness of both discharges and resists. They are particularly suitable for printing on Naphtol AS grounds and can also be used alongside stabilised azoic combinations, solubilised vat dyes, Phthalogens, and resin-bonded pigments, nine patterns illustrating these and other styles being included in the card. Fastness figures on cotton include—

	Light	Washing (c) (203°F.)
Permafix Yellow 4G	5-6	4-5
Permafix Brilliant Blue 3GL	6-7	4-5

SIRIUS SUPRA BLUE GRL—This direct dye gives blues of good fastness to light and comparatively good fastness to washing on cellulosic fibres. It has very good solubility, good levelling properties and is stable to prolonged boiling. Dyeings become greener when crease-resisted but their light fastness if hardly affected. After-treatment with Levogen WW, FWM or HW considerably improves their fastness to wet treatments. Type 8002 of this dye is suitable for white and coloured discharges using a special alkaline discharge. Fastness figures on cotton include—Light 6, washing (a) (40°C.) 2-3, perspiration 2-3.

BLANKOPHOR CE—A fluorescent brightening agent for cellulosic fibres and polyamide fibres. It is a derivative of stilbene disulphonic acid, is stable in hypochlorite and chlorite bleach baths and gives a neutral to slightly greenish effect. The brightening obtained has good resistance to mild washing on cellulosic fibres but not to severe washing at the boil; on nylon the resistance is very good. Stability to light is very good for this type of product.

Farbwerke Hoechst A.G.

OFNA-PON ASN—This is an anionic colloid preparation for use in padding liquors when applying Naphtols. A particular advantage is that impregnation baths containing this product are stable in presence of up to 30 g./l. common salt, even after cooling or prolonged storage. Its presence in padded and dried materials markedly increases their re-wet ability by cold and acid solutions. For padding, 8-10 g./l. of Ofna-pon ASN should be added and 2-3 g./l. when impregnating on the jig or in long liquors, with 30% of the original quantity added as replacement in the latter case.

The Geigy Co. Ltd.

IRGAVEL DC—A substantive softening and antistatic agent for use in dry cleaning and applied from the rinse solvent by exhaustion.

PIGMENTS AND PIGMENTS—This booklet outlines the general chemical structure and properties of lakes, metal toners, metal-complexes, pigmentary colours (metal-free organic compounds devoid of groupings imparting water-solubility), dye-complexes, and phthalocyanine pigments. Typical structural formulae are given and a number of black and white photographs of plant and equipment are included.

J. R. Geigy A.G.

REACTONE DYES IN PADDING—This card describes the application of the new range of Reactone reactive dyes to cellulosic fibres by pad-fixation methods. All twelve members of the range recommended for printing are recommended for this purpose also. Reactone Navy Blue GRL is recommended for use as a blue and a navy but Reactone Black RL is shown as a grey only. The Reactones are very soluble (> 100 g./l. at 50°C. in all cases), show no substantivity for cellulose in absence of electrolytes and are marketed in highly concentrated form to reduce to a minimum the effect of electrolyte added in standardising. Neutral solutions are stable almost indefinitely. Solutions containing 20 g./l. soda ash are unaffected after standing for 30 days at 20-25°C. and fabric padded with such solutions and stored for this length of time at room temperature show no reduction in depth when fixed. The usual thorough preparation of the cloth is essential. To promote adequate diffusion into the fibre, especially when using Turquoise Blue FGL and producing deep dyeings with other dyes, the padded material should be allowed to lie for an hour. Urea (up to 200 g./l.) is added to the pad-liquor to improve fixation. It also effectively counteracts the effect on substantivity of the added electrolyte, including Reserve Salt G (sodium *m*-nitrobenzene sulphonate) included in many recipes. It also promotes

migration during intermediate drying and sodium alginate may be added as a migration inhibitor, but the effect of this in reducing the rate of dye diffusion must be watched. Recommended fixation methods include (1) on the jig in concentrated salt solution with alkali; (2) dry heat at 140–160°C. for 6–4 min. for cotton and at 130–150°C. for 5–4 min. for regenerated cellulose; (3) steaming at 102–104°C., Reserve Salt G being added to the pad-liquor. Washing-off must be thorough, the sequence, cold rinse-hot rinse-soaping at the boil-hot rinse-cold rinse, being recommended, particular care being required when using the Turquoise Blue FGL. The card contains dyeings in three depths of each dye and 16 mixture dyeings from bulk production. Fastness figures show the expected very good fastness of these dyes to repeated washing and good to very good fastness to light. The quality of the pattern card itself is excellent.

Imperial Chemical Industries Ltd.

CHROMASTRAL RED RS—This is a co-precipitated blend of a molybdated scarlet chrome with a red monoazo pigment and combines the latest advances in darkening-resistant scarlet chromes and in solvent-resistant monoazo pigments. It has high opacity, good solvent and heat fastness, and good weathering fastness.

MONOLITE FAST YELLOW FRS—This is a pure pigmentary colour having excellent stability to heat and solvents and excellent weathering fastness even in very pale depths. It is of particular interest in the coloration of high-quality industrial finishes of all types, especially car lacquers. It is also of interest in decorative paints, clear lacquers, in certain speciality printing inks and for colouring plastics where high processing temperatures are not required.

MONOLITE MAROON GS—This is a metal complex of a monoazo pigment giving golden maroons in full depth, both as a reduction with white pigment and in combination with metallic aluminium. Its colour, stability, resistance to bleeding and high weathering fastness makes it of interest in industrial finishes, and in paints and printing inks based on drying oils.

PROCION/ALKALI MIXER—An efficient and relatively simple automatic pre-mixing device for this purpose has been devised in the laboratories of the Dyehouse Department and is manufactured under licence by the Horsfall Engineering Co. Ltd. of Ironmonger Lane, Oldham, Lancs.

PROCION BRILLIANT ORANGE 2R—This homogeneous reactive dye gives brilliant reddish oranges when dyed on cellulosic fibres. Dyeings are much redder in hue than those given by Procion Brilliant Orange G and have much better fastness to hypochlorite bleach liquors. Their fastness to light is a little lower also. Its affinity for cellulose is lower than that of Brilliant Orange G and thus removal of unfixed dye is easy and the risk of staining and marking-off in washing is reduced. It is suitable for application, alone and in mixtures, by batchwise dyeing and also by semi-continuous and continuous methods. Fastness figures on cotton include—Light (Wilmslow) 4, soda boil/hypochlorite bleaching 4, washing (repeated severe) 5 times at 100°C., 4–5.

PROCION BRILLIANT PURPLE H3R—This homogeneous copper-complex reactive dye gives reddish violets of very good fastness to light, washing, and hypochlorite on cellulosic fibres. It is very soluble and thus eminently suitable for pad-application. It can be applied by all the methods generally recommended for "H" brand dyes and by hot batchwise dyeing techniques, showing good colour yield and good building-up properties on regenerated cellulose rayons. Fastness figures on cotton include—Light (Wilmslow) 6–7, perspiration ("natural") 4, washing

(repeated severe, 5 times at 100°C.) 5, soda boiling/hypochlorite bleaching 4.

PROCION SCARLET HR—This new homogeneous reactive dye gives bright scarlets of moderate to good light fastness and very good fastness to washing on cellulosic fibres. It is more soluble than Procion Scarlet H3G and gives dyeings which are bluer in hue. It can be applied by all the methods recommended for Procion "H" dyes, the most suitable being the pad (carbonate)-dry-steam and the pad (alkali, urea)-dry-bake techniques. On the Pad-Roll machine and by the pad (caustic)-batch (cold) method weaker dyeings are obtained and in the latter case the stability of the pad liquor is poor. Fastness figures on cotton include—Light (Wilmslow) 3, washing (repeated severe, 5 times at 100°C.) 4–5, hypochlorite 2.

DISPERSOL AFB—This is a retarding agent for use when applying basic dyes to acrylic fibres. Initial dye absorption is restrained to a marked extent and a much more rapid rise in dyebath temperature from cold to 100°C. is possible. Further, unlike cationic assistants, it does not exhaust rapidly on to the fibre and a further risk of uneven dyeing is thus eliminated. Some change in hue may occur when it is used but this is no problem if small-scale matchings are made. Fastness properties are unimpaired. The presence of excessive amounts of Dispersol AFB reduces the maximum depth of colour obtainable. A small number of basic dyes have been found to be precipitated in the dyebath and laboratory tests should be carried out by the user before use.

TECHNICAL INFORMATION LEAFLET—Dyehouse No. 546. Combined Dyeing and Finishing of Cotton Piece Goods by the Procion-Resin Process—Procion reactive dyes will react with basic groups of resin precondensates under the acidic conditions of curing during polymerisation, and by using this principle it is possible to dye and resin-finish cotton cloth. The Procion-Resin process is recommended for use with a number of the resin precondensates and a number of important economic and technical advantages are claimed including saving in overall costs and improved colour yield. No major changes in finishing practice are required. The Procion dye and a special catalyst, Procion-Resin Catalyst A, are added to the pad-liquor and the normal sequence, pad-dry-bake-wash-off, is followed. The fastness of the dyeing produced depends on the durability of the resin finish and control of the finishing process is thus even more important than when the finish is applied to previously dyed goods. A selection of suitable resin types are listed and a range of sixteen dyes is given which will meet the stated minimum fastness requirements in all depths. The fastness to water of full-depth dyeings using dimethylol cyclic ethylene urea deteriorates on storage in acid atmospheres and consequently on pale dyeings should be produced with dyes giving the highest light fastness. Procion Brilliant Blue H7G, in general, gives dyeings of inferior fastness properties compared with the other selected dyes but with "Fixapret" CP in particular the results are comparable with the standard achieved for this colour using normal dyeing methods followed by resin finishing.

Sandoz A.G.

PALETTE No. 4—This issue contains the following articles. "Colour as Clothing: The Role of Colour in the Body Painting of Primitive Peoples" by J.-J. Pittard; "Batch" by A. Steinmann; "Colour in Chemical Analysis" by S. A. Jimeno; "Viscosity and double Refraction during Flow" by R. Signer and "Terylene and some of its Derivatives" by H. Wahl. The first two are illustrated by some striking colour photographs.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Dyeing or Wet Processing of Hanks

Longclose Engineering Co. BP 832,498 (1 Sept 1955)

The hanks are suspended from sticks. Sticks are also passed through them at or close to their lower ends and parallel to the top sticks. These lower sticks project and the outer hanks of each row are coupled together by longitudinal rods to form a frame which is suspended from the lid frame by a swinging support. This maintains the hanks in an orderly and extended condition during treatment. C.O.C.

Shearing Machines for Yarn, Cord, and Twine of Long Fibres such as Sisal and Manilla Hemp

James Mackie & Sons BP 833,469 (3 Dec 1956)

Wet Treatment of Fabrics

H. Brugman BP 831,245 (Holland 27 June 1957)

Fabric passes continuously through one or more vessels each containing a vibration member connected to a vibrator. This member consists of plates corrugated horizontally, i.e. perpendicular to the direction of oscillation of the vibrator. The plates are interconnected in pairs so as to be check-patterned viewed in cross section. They are suspended from a vertically oscillatory vibrator which imparts to them longitudinal oscillations at right angles to the corrugations. At the bottom of the plate there are guide faces to direct the fabric which is led down the container on one side of the vibration member and up on the other side. C.O.C.

Pressure Dyeing Machine for Warps, Cloth, etc.

V. T. Fahringer USP 2,905,522 (22 April 1954)

The material passes through a pressure vessel having opposed openings slightly greater than the thickness of the material. Dye liquor is forced into the vessel under pressure and is maintained above atmospheric pressure by there being a liquid seal on the outside of each of the openings in the vessel. The fluid forming the seal is heated to at least the boil under atmospheric pressure. C.O.C.

Variable Camber Bar

J. F. Collier & Co. BP 832,106 (16 Dec 1957)

A variable camber bar consists of a number of hollow sections. One end of each section has a reduced cross section so that it can fit into the next. The reduced end tapers only over part of their peripheral surface. The shoulder at each reduced end makes, when the bar is straight, an angle relative to the next section corresponding to the angle of taper. A rod passes through the sections and can be tensioned so as to cause the camber bar to bow, the extent of this bow being determined by the degree of tension. C.O.C.

Tensioning Web or Strip Material

Spooner Dryer & Engineering Co. BP 833,525 (9 Feb 1957)

Heating Moving Webs

Spooner Dryer & Engineering Co. BP 830,989 (14 Aug 1957)

Modification of BP 809,821 (J.S.D.C., 75 (1959) 259). Cloth, paper or the like is conveyed horizontally through a chamber in which streams of a hot gas impinge perpendicularly on to it or at a substantial angle thereto. This heats the cloth or other material to or near to polymerisation or vulcanisation temperature. It then passes through a second chamber where a gas is circulated in a closed circuit at or above polymerisation or vulcanisation temperature. The time taken for the cloth to pass through the second chamber is long enough for complete polymerisation or vulcanisation to occur. C.O.C.

Automatic Regulation of the Continuous Application of Specified Amounts of Solids or Liquids to a Moving Web

British Cotton Industry Research Association BP 830,836 (23 April 1958)

The material to be applied is supplied to a reservoir from which it is applied to the web at a rate predetermined by the

speed of movement of the web and the amount of material to be applied per unit length of the web. After application of the material the web passes through a pair of squeeze rollers whose pressure is altered by deviation of the actual rate of application of the material from the prescribed rate. This has the effect of correcting the deviation. C.O.C.

Continuous and Uniform Distribution of Fine Powders on Surfaces

Technoplast—Spindler

BP 832,523 (Germany 31 Aug 1955)

The powder used is of such particle size that it is capable of being electrostatically charged to such an extent that the particles' adhesion to other bodies is sufficient to support their own weight. It is applied through one or more rapidly moving screens having on their upper surfaces freely movable elements which carry out a secondary movement under the influence of the screen movement. This causes screening to be nearly instantaneous so that the powder leaves the screen in the manner of a liquid although it has been strongly charged electrostatically by friction with the moving elements. C.O.C.

Applying Particles to a Surface in Predetermined Patterns

Bird & Son

USP 2,905,569 (3 Jan 1955)

Apparatus which deposits non-liquid particles in areas of predetermined shape with great accuracy and sharp definition, and with great freedom from carry-over of particles into areas in which they are not desired. C.O.C.

Drying Machine for Tubular Fabrics

Fitch Textile Co. BP 832,779 (U.S.A. 18 July 1955)

A simplified machine to continuously dry the fabric to desired width more rapidly and with less use of power per unit length of fabric than required by those hitherto available. C.O.C.

Printing and Dyeing File Fabrics (VIII p. 461)

Flexible Material Made Up of Several Layers (XI p. 466)

II—WATER AND EFFLUENTS

PATENTS

Water Purification

John Thompson-Kennicott BP 830,964 (13 Jan 1955)

Reduction of the amount of organic matter both in solution and in colloidal suspension in the raw water is effected by adding $KMnO_4$ in amount insufficient to completely oxidise the organic matter, so that oxides of Mn and/or their hydrates are precipitated. The water is then passed through a bed of porous mineral granules which have been successively treated with solutions of $MnSO_4$ and $KMnO_4$ so as to deposit MnO_2 in the pores of the mineral. It may then be passed through a bed of ion exchange resin to remove mineral salts. In some cases the ion exchange bed may also filter out the precipitate from the $KMnO_4$ treatment in which case the passage through the bed of mineral granules may be omitted. C.O.C.

Separation Tanks for Effluents

J. F. Terrett & C. R. Strutt BP 833,605 (12 Nov 1957)

Tanks especially designed for effluents from which fat, oils, and solids have to be separated. C.O.C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Epoxide Wash-Wear Finishes for Cotton

G. Galligan, A. M. Sookne, J. T. Adams, H. Guest, and G. H. Lourigan

Textile Research J., 30 (March 1960) 208-222

A wide range of epoxides is available for finishing cellulosic fibres, their properties vary so greatly that they differ from one another just as much as they do from other commercially available resins. The properties they impart to cotton also vary greatly as regards colour and loss in strength as does the rate of hydrolysis of the epoxide in the liquor. Thus it is unreasonable to consider

C.O.C.

C.O.C.

C.O.C.

C.O.C.

$$\text{Hal}_n - \text{C}_6\text{H}_4 - \text{NH} - \text{C}(\text{X}) = \text{Y}$$

C.O.C.

$$\text{H}_2\text{N}-\left[\begin{array}{c} \text{X} \\ \parallel \\ \text{P}-\text{NH}- \\ | \\ \text{N} \text{Y}^1 \text{Y}^2 \end{array}\right]_n \text{H}$$

C.O.C.

C.O.C.

C.O.C.

C.O.C.

C.O.C.

The quant. relations between the degree of deviation from coplanarity of the mol. configuration and the ultraviolet absorption spectrum as well as the quantities such as the extra-resonance energy and the π -bond orders are determined for stilbene-type cpd. by calculations based on the simple LCAO mol. orbital method. It is inferred that the most probable configuration of *trans*-stilbene in soln. is not significantly different from that in the cryst. state, viz. planar or nearly so. The interplanar angles of *cis*-stilbenes are estimated at $\sim 28^\circ$. The solvent effects of benzene on the spectra of *trans*-stilbene and its *p*-methoxy- and *p*-nitro-deriv. viz. a large

bathochromic displacement of the conjugation band and a remarkable redistribution of intensity among its vibrational bands, are discussed.

VI—Triphenylethylene, Tetraphenylethylene, and Tolan

Ibid., 389-396

The method of V is applied to the title cpd., and the most probable configurations of tri- and tetra-phenylethylene are inferred to be the ones in which all the phenyl groups are rotated out of the plane of the ethylene bond. The calc. and observed wavelengths of the bands for tolan agree fairly well, and its spectrum in the cryst. state resembles that in n-heptane soln.

VII— α -Substituted and α,α' -Disubstituted Stilbenes

Ibid., 396-405

It is especially noteworthy that the *trans*- show the conjugation band at a shorter wavelength than the *cis*-isomers. This fact, contrary to expectation, is considered to indicate that in these particular cpd. the *trans*- are less conjugated than the *cis*-isomers, and that the angle of twist of the single bond (interplanar angle) is larger in the *trans*-isomers.

VIII—Stilbene Derivatives with Methyl Groups on the Benzene Nuclei

Ibid., 406-410

Data are discussed for 4-methyl- and 4,4'-dimethylstilbene, 4,4', α,α' -tetramethylstilbene (*cis*- and *trans*-isomers), 2,4,6-trimethyl- and 2,4,6,2',4',6'-hexamethyl-*trans*-stilbene.

IX—*p*-Phenylstilbene, *p,p'*-Diphenylstilbene, and their Related Compounds

Ibid., 410-417

Data and LCAO calculations are reported for *p*-phenylstilbene, 1-phenyl-2-(2-fluorenyl)-ethylene, and *p,p'*-diphenylstilbene and its α,α' -dimethyl deriv. The red-shifts of the conjugation bands when the solvent is changed from benzene to n-heptane and their correlation with the spatial configurations of their respective mol. are discussed.

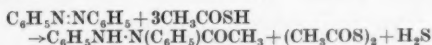
H.H.H.

Effect of Thioacetic Acid on Anils and Azobenzene

B. M. Mikhailov and I. S. Savel'eva

Izvestiya Akad. Nauk S.S.S.R., Otdel khim. nauk, No. 7 (July 1959) 1304-1306

Benzophenone anil and acetophenone anil react with thioacetic acid (TA) with the formation of acetanilide and thiobenzophenone (TP), or acetophenone respectively. In the presence of oxygen, TP and TA undergo conjugated oxidation with formation of benzophenone, diacetyl-disulphide, diacetylsulphide, hydrogen sulphide, and sulphur. On heating, azobenzene reacts quantitatively with TA—



Experimental details are given.

G.J.K.

Condensation of Naphthalic Anhydride and its Derivatives with Arylamines. IV—Azo Dyes from Aminophenylimides of Phthalic, Naphthalic, 4-Nitro- and 4-Aminonaphthalic Acids

E. S. Khotinskii, R. M. Matskevich, and B. M. Krasovitskii

Uchenye Zapiski Kharkov Univ., 71 (1956), 155-163

Referat. Zhur. Khim., 1957, Abstr. No. 30588

Chem. Abs., 54 (25 Feb 1960) 3959

m- and *p*-Phenylenediamine condensed with phthalic, naphthalic, and 4-nitronaphthalic anhydrides gave the *m*- and *p*-aminophenylimides which were diazotised and coupled with H, R, and Schäffer acid giving monoazo dyes for wool. Reduction of the nitro groups in the aminophenylimides of 4-nitro-phthalic and -naphthalic acid gave diamines from which diazo dyes were prepared.

E.S.

Azo Dyes by Coupling of Diazo compounds with Sulphonic Acids of Phenols, Naphthols, Naphtylamines and Aminonaphthols

M. S. Zhedek

Zhur. priklad. khim., 33 (March 1960) 707-711

31 Azo dyes were prepared from diazotised 4,4'-diaminodiphenyl sulphonic acid and sulphonated phenols, naphthols, naphthylamines, and aminonaphthols. The chemical and dyeing properties of these compounds were

investigated. They dye wool, silk, cotton, and viscose giving results fast to washing, light, perspiration, and heat.

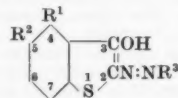
L.S.L.

Studies in Azo Dyes. Part I (Monoazo Dyes from Thioindoxyls)

P. C. Dutta, A. K. Sen Gupta, B. P. Bose, and S. K. Roy

J. Indian Chem. Soc., 37 (Jan 1960) 50-52

Thioindoxyl and its 5-phenyl- and 4,5-benzo-derivatives were coupled with diazo compounds to give monoazo dyes—



(R^1 , R^2 = H, or together = benzo, or R^2 = phenyl; R^3 = aryl which may contain NO_2 , SO_3H , etc., groups), whose λ_{max} values were determined. Presence of the 5-phenyl group tends to a bathochromic effect; the 4,5-benzo group has little effect on the hue.

E.S.

Absorption Spectra of Some Monoazo Acid Dyes

N. A. Boguslavskaya, B. M. Bogoslovskii, and B. M. Yavorskii

Zhur. obshch. khim., 30 (Jan 1960) 112-115

24 Monoazo acid dyes were synthesised using 6 isomers of α -naphthylamine sulphonic acid which were diazotised and coupled with four different coupling compounds: α - and β -naphthols and α - and β -naphthylamines. The effect of structural changes on absorption spectra of dyes was examined at 220-700 $\text{m}\mu$. Change in the position of the sulphonic group does not affect the spectrum of α -aminosulphonic acid dyes but with β -amino-2- and β -amino-8-sulphonic acids there is 25-50 $\text{m}\mu$. difference in the displacement of absorption peak. The hydroxy group has more effect on the position of the absorption peak than the amino group. Changing OH groups from α - to β -position displaces λ_{max} towards the longer wavelength.

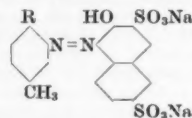
L.S.L.

Relationship between Dye Constitution and Properties of Dyed Material. II—Fastness of Acid Monoazo Dyes—Derivatives of Toluene-azobenzene on Wool

B. I. Stepanov and M. V. Zakharova

Tekhnol. tekstil. prom., No. 2 (9) (1959) 117-124

13 Monoazo acid dyes, of formula—



(R = H, Cl, alkoxy, or phenoxy) were applied to wool (3.0 and 0.5%) and the fastness to water, soap, perspiration, dry and wet rubbing, decatising, carbonising, and light tested and the results tabulated. Presence of the ether grouping improves wet fastness properties of these dyes; alkoxy being in general superior to phenoxy groups, and the effectiveness within the alkoxy series ($-\text{OCH}_3$ to $-\text{OC}_8\text{H}_{17}$) increasing with increasing carbon content. The light-fastness of 3% dyeings is slightly improved by phenoxy grouping; that of 0.5% dyeing is decreased throughout.

G.J.K.

Structure and Rearrangement of Diazo Compounds XII—Investigation of Spectra of Some Aromatic Diazo Compounds

B. A. Porai-Koshits and B. V. Passet

Zhur. obshch. khim., 30 (Jan 1960) 286-290

Aqueous solutions of diazo compounds display selective absorption in the ultraviolet region. In acid and alkaline solutions various absorption bands were observed, whilst at pH 7-9 the absorption curves of diazo compounds possess isobestic points. Presence of isobestic points suggests that in aqueous solution diazo compounds at equilibrium contain only diazo anions and diazo cations. Neutralising the diazo cation with alkali is not a first order reaction which indicates that the second stage, i.e. dissociation of diazo acid and anion formation, occurs at a great speed. Neutralisation of the diazo anion is a first order reaction. Aqueous solutions of all the investigated aromatic diazo compounds observe Beer's and Lambert's Laws.

L.S.L.

Separation Component in the Synthesis of Organic Dyes. XI—Synthesis of Disazo Dyes from Tetrazotised 1,5- and 1,4-Diaminoanthraquinones

I. Reichel and W. Schmidt *Acad. rep. populare Romine, Studii cercetari chim.*, 7 (1959) 61–71
Chem. Abs., 54 (25 Feb 1960) 3338

1,5-Diaminoanthraquinone was tetrazotised in nitrosyl-sulphuric acid at 40°C., and coupled with various phenols, naphthols, etc., e.g. with salicylic acid which gave a brown for cotton. 1,4-Diaminoanthraquinone was tetrazotised in a mixture of acetic acid and conc. HCl by adding isoamyl nitrite at room temperature, and gave dyes deeper in colour than those from the 1,5 compound. E.S.

Utilisation of 1,4-Naphthoquinone

VII—Synthesis of 2-Arylamino-1,4-naphthoquinones

A. Inoue, N. Kuroki, and K. Konishi

Yuki Gosei Kagaku Kyokaiishi, 17 (1959) 711–713

1,4-Naphthoquinone (3.95 g.) and Cu acetate (5) were dissolved in methanol (50 c.c.), stirred at 60°C. for 30 min. while introducing air (16 c.c./min.) and a solution of *p*-chloroaniline (0.03 mole) in methanol (40 c.c.) was added. Air was bubbled through the mixture while stirring for 3 hr. The mixture was then cooled and filtered. The resulting mass was dissolved in aqueous alkaline hydro-sulphite at 60°C. and filtered to give a 73.6% yield of 2-(*p*-chlorophenylamino)-1,4-naphthoquinone which dyes wool and silk dark red. The following 2-(RNH)subst.-1,4-naphthoquinones were also prepared (R, % yield, colour on wool, silk, acetate, Vinylon, and Amilan are given: (C₆H₅, 52.7, red, dark red, —, —, —; 1-naphthyl, 20.1, brown, brown, —, —, —; *o*-methoxyphenyl, 7.53, dark red, dark red, reddish yellow, dark red, dark red; *p*-methoxyphenyl, 94.0, light yellow, light yellow, yellowish red, dark red, dark red; *p*-(1,4-naphthoquinon-2-ylamino)-phenyl, 83.0, dark violet, dark brown, —, —, —; 4'-(1,4-naphthoquinon-2-ylamino)-4-biphenyl, 74.1, brownish violet, brownish violet, —, —, —; 3,3'-dimethoxy-4-(1,4-naphthoquinon-2-ylamino)-4-biphenyl, 81.7, dark violet, dark violet, —, —, —; *p*-hydroxyphenyl, 52.8, —, —, reddish brown, dark red, dark reddish violet; CH₃, 70.7, —, —, yellowish orange, dark orange, orange; CH₃CH₂OH, 53, —, —, yellowish orange, dark yellow, light yellow.

VIII—Derivatives of 1-Phenylazo-2,4-dihydroxy-naphthalene

Ibid., 714–716

Chem. Abs., 54 (10 March 1960) 4504

Hydroxy-1,4-naphthoquinone (I) is prepared from 1,4-naphthoquinone, 2,3-Dichloro-4-naphthoquinone (0.06 mole) and NaNO₂ (0.2 mole) are dissolved in methanol (75 c.c.) and water (105 c.c.), stirred at 80°C. for 3 hr., filtered hot and the filtrate cooled. The precipitate is dissolved in warm water, acidified with HCl, cooled, and the precipitate washed with cold water to give a 77.8% yield of the yellow 2-hydroxy-3-nitro-1,4-naphthoquinone (II). The yellow 2-hydroxy-3,5(or 8)-dinitro-1,4-naphthoquinone (III) was similarly prepared in 44.5% yield. 2,3-Dichloro-4-naphthoquinone (0.1 mole) was dissolved in hot 5% aq. NaOH (160 g.), filtered hot and acidified with HCl to give 91.1% 2-hydroxy-3-chloro-1,4-naphthoquinone (IV). 2-Hydroxy-3-chloro-5-nitro-1,4-naphthoquinone (V) was similarly prepared in 92.1% yield. I (0.02 mole) in aqueous methanol was added to phenylhydrazine (0.03 mole) in methanol (15 c.c.), stirred at room temperature for 3 hr. and water added to give a 96.2% yield of 1-phenylazo-2,4-dihydroxynaphthalene (VI) (λ 437 m μ). The following 1-phenylazo-2,4-dihydroxy-3-R¹-5(or 8)-R²-naphthalenes were similar [starting material, R¹, R², % yield and λ in m μ]: II, NO₂, H, 70.9, 434; III, NO₂, NO₂, 21.3, 440; IV, Cl, H, 68.3, 451; V, Cl, NO₂, 96.2, 448. I (0.005 mole) and 2,4-dinitrophenylhydrazine (0.005) were dissolved in ethanol, 2 drops conc. H₂SO₄ added, stirred at 80°C. for 4 hr., cooled and recrystallised from ethanol to give 98.4% 1-(2',4'-dinitrophenylazo)-2,4-dihydroxynaphthalene. The following 1-(2',4'-dinitrophenylazo)-2,4-dihydroxy-3-R¹-subst.-5(or 8)-R²-subst.-naphthalenes [starting material, R¹, R², % yield, λ in m μ]: II, NO₂, H, 72.5, 442; III, NO₂, NO₂, 50.2, 450; IV, Cl, H, 85.4, 444; V, Cl, NO₂, 43.1, 450. Their affinities for acetate, Vinylon, and Amilan are given. In general they yield clear

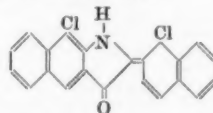
dyeings but those prepared by coupling with 2,4-dinitrophenylhydrazine have slightly less affinity for the fibres. Fastness to light is not good except for several dyeings on Vinylon.

IX—Algol Brilliant Green BK (C.I. 73840) and Its Homologues

Ibid., 773–776

Chem. Abs., 54 (10 March 1960) 4537

C.I. 73840 and its ethoxy (I) and propoxy (II) analogues were synthesised and their properties compared. Dry HCl gas (5 g.) was absorbed in methanol (45), SnCl₄ (14) added and the mixture refluxed with 1,4-naphthoquinone (7.9) for 2 hr., poured into water and the precipitate recrystallised from C₆H₆ to give 1,4-naphthohydroquinone monomethyl ether (5.2) (III). 57.6% and 46.5% yields were similarly obtained of the monoethyl ether (IV) and monopropylether (VI) respectively. 2-Naphthylamine (28.6 g.), powdered S (0.4), CS₂ (22.8) and methanol (100 c.c.) were refluxed 5 hr. to give NN'-di-2-naphthyl urea (29.4 g.) (VI). VI (29.4 g.) was mixed with methanol (150 c.c.) and basic Pb carbonate [prepared from Pb(NO₃)₂ (35.8) and Na₂CO₃] and treated with NaCN (6.2) dissolved in water (50 c.c.) to give 2-C₁₀H₇NHC(CN):NC₁₀H₇-2 (VII). VII (9.6 g.), chlorobenzene (120 c.c.) and SO₂Cl₂ (7.9 g.) were heated together, then treated with AlCl₃ (12), poured on to ice, neutralised with aq. Na₂CO₃ and treated with steam to give VIII (11.5 g.).



After further purification VIII (2.3 g.) in nitrobenzene (30 c.c.) was treated with Br (2.2 g.), kept at room temperature for 1 hr., 45–50°C. for 8 hr. and then 100–105°C. for 9 hr. and washed with ligroin to give α -bromo-1-chloro-2,3-naphthistatin (2.6 g.) (IX). IX (1.6 g.), chlorobenzene (25) and PCl₅ (1.2) were heated at 115°C. for 3 hr., then treated with III (0.0064 mole) in chlorobenzene (20 g.) at 60°C. for 30 min. and 100°C. for 30 min., and washed with chlorobenzene and methanol to give 73% C.I. 73840. Similar reaction of IX with IV and V gave 70.9% I and 73% II respectively. Hues and fastness of C.I. 73840, I and II were compared. Vattability and affinity for the fibre decreased with increase in numbers of C atoms in the alkoxy group. Dyeing trials of the sulphonic acid esters of these dyes on wool and cotton indicated that the hypsochromic effects slightly decreased with increase in numbers of C atoms in the alkoxy group this being more marked in the case of wool than cotton. Differences in the alkoxy group have negligible effect on the fastness to light. C.O.C.

Reactions of β -Naphthol with Thioglycolic Acid and Other Mercaptans

F. M. Furman, J. H. Thelin, D. W. Hein, and W. B. Hardy
J. Amer. Chem. Soc., 82 (20 March 1960) 1450–1452

Thioglycolic acid (I) reacted with β -naphthol (II) in equimolar amounts in presence of NaOH in ethylene glycol by heating for 23 hr. at 145–150°C. β -Hydroxy-1-naphthalene acetic acid was obtained in 89% yield. 1.0–1.5 molar equiv. NaOH gave optimum yields. Treatment of (II) at 110–120°C. with various mercaptans for 16 hr. in presence of acid catalysts (e.g. *p*-toluene sulphonic acid) without solvent gave β -naphthylsulphides thus providing a useful synthetic route to alkyl and aryl naphthyl sulphides. Mercaptans used were of formula HSR (% yield) (R = —CH₃ (99%), —C₂H₅ (68%), —(CH₂)₃CH₃ (22%), —(CH₂)₁₁CH₃ (79%), —CH₂COOH (87%), —C₆H₅ (43%). α -Naphthol, 6-bromo-2-naphthol, and 1,5-naphthalenediol reacted similarly with (I) to give corresponding naphthylthioacetic acids. Possible mechanisms for above reactions are discussed briefly. F.J.

Electronic Structure and Physicochemical Properties of Azo Compounds. VII—Absorption Spectrum and Basicity of the *p*-Trimethylammoniumphenyl-azoazulene Cation

F. Gerson, J. Schulze, and E. Heilbronner
Helv. chim. Acta., 43 (15 March 1960) 517–523

Conclusions drawn previously (cf. *J.S.D.C.*, 76 (1960), 46) respecting the influence of —M and +M active

substituents on the spectra of 23 deriv. of phenylazoazulene are now supported by the spectroscopic properties and basicity of the cation of *p*-trimethylammoniumphenylazoazulene chloride (prepared by coupling the corresponding trimethylammoniumphenyldiazonium ion with azulene). In this particular case the *p*-trimethylammonium substituent exerts a strong +M and no -M effect. The structure assigned previously to the protonated *p*-dimethylaminophenylazoazulene (cf. *loc. cit.*) is verified. A σ_p -value of 0.66 ± 0.03 for the trimethylammonium substituent is reported. H.H.H.

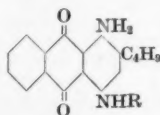
New Intermediates and Dyes. VIII—1,4-Di-substituted Derivatives of 2-Butylanthraquinone

A. T. Peters, Jr. and A. T. Peters

J.C.S., (1960) 1125–1130

2-Butylquinizarin (I) is obtained in good yield by refluxing 1,4-diamino-2-n-butylanthraquinone (II) with hydrosulphite in aqueous alcoholic NaOH, followed by H_2SO_4 oxidation, or by reaction under N_2 of either leucoquinizarin or 1,4-diaminoanthraquinone with butyraldehyde, followed by aeration. I with NH_4OH gives a mixture of II and mono-amidated products.

1-Amino-4-bromo-2-n-butylanthraquinone (Part VII, *J.C.S.*, 230 (1959) 3928) condensed with amines NH_2R gave violet to blue dyes.



Condensations were effected under reflux or under closed conditions in presence of Na and Cu acetates and all products were purified by chromatography on alumina. For $R = Alk$, dyes were weaker than $R = H$, but bluer. $R = n-C_4H_9$ was the strongest dyeing on acetate, nylon, and Terylene, and $R =$ branched chain alkyl gave only weak dyes. $R =$ hydroxyalkyl, dyes were stronger and redder than $R = Alk$, and $R =$ methoxyalkyl gave stronger, brighter, and redder dyes than any of the above, and also imparted excellent light fastness. $R =$ cyclohexyl and benzyl gave only weak violets on acetate and nylon and a moderately weak blue on Terylene. $R = Ar$ resulted in greenish blues of rather low affinity, but substitution of aryl by hydroxyalkyl gave dyes of excellent affinity on all fibres and good all-round fastness, especially to light and washing. Generally R was bathochromic in order p -hydroxyalkylphenyl $>$ p -tolyl $>$ phenyl $=$ m -tolyl $>$ o -tolyl $>$ alkyl $>$ hydroxyalkyl $>$ methoxyalkyl $>$ cyclohexyl $>$ benzyl $>$ H and affinity was methoxyalkyl $>$ hydroxyalkyl $>$ $H =$ hydroxyalkylphenyl $>$ n -alkyl $>$ isoalkyl $>$ benzyl $>$ cyclohexyl $=$ phenyl $=$ tolyl. A.T.P.

Constitution of the Red and Colourless Forms of Quinolylmethanes

G. Scheibe and W. Riess

Chem. Ber., 92 (1959) 2189–2198

Chem. Abs., 54 (1960) 3423

The isomerism of di-(2-quinolyl)methane (I) and tri-(2-quinolyl)methane (II) was investigated by spectroscopic methods. Comparison of the coloured forms of I and II with di-(2-quinolyl)amine (III), quinoline red (IV) and a pseudo isocyanine (V) indicated that I and II contained a H bridge, which caused electrosomerism.

Also recorded were the u.v. absorption spectra of the colourless and red forms of I and II, quinoline, 1-methyl-2-quinolone (2-quinonyl methide) in alcohol at $20^\circ C$, of III in 2:1 methylcyclohexane-isopentane at $20^\circ C$, and in alcohol, and of 2-aminoquinoline in alcohol at $20^\circ C$. Numerous absorption and fluorescence polarisation spectra are also given. A.T.P.

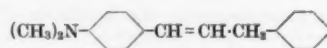
A Quinopyrylium Dye

A. Treibs and H. Bader

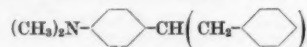
Chem. Ber., 90 (1957) 789–793

Chem. Abs., 54 (1960) 3402

Reaction of *p*-dimethylaminobenzaldehyde (I) and phenylmethylketone (II) gave a variety of products dependent on the conditions used. Thus, by refluxing for 2 hr. in NaOH (0.05 mole) and alcohol (150 ml.), 0.1 mole I and 0.05 mole II gave 60% of (III)—



and 0.05 mole I and 0.1 mole II gave 30% of IV—



III and IV were interconvertible by reaction with excess of either I or II respectively. IV boiled in a mixture of acetic acid and acetyl chloride was oxidised and dehydrated to give 35% of 2,6-diphenyl-4-(*p*-dimethylaminophenyl)pyrylium chloride, deep violet. Quinopyrylium is the name suggested by the authors for 4-(*p*-aminophenyl)pyrylium ions. A.T.P.

Phthalogens in the Framework of the Phthalocyanines

J. Eibl

Teinture et Apprêts, 56 (Feb. 1960) 6–24

The historical background of phthalocyanine development is described, together with a general description of the various types of phthalocyanine dyes at present employed in commerce. The author discusses in detail, constitution, properties, synthesis and structures of Phthalogen dyes, together with all relevant reactions in their formation and use. The two classes of Phthalogens, viz. metal-free amino-imino-isoidolenines, e.g. Phthalogen Brilliant Blue IFGG, and the heavy-metal-containing polyisoidolenines, e.g. Phthalogen Brilliant Blue IF2GK, are treated separately. Comparison of the properties of both classes indicates the different types of colouring processes possible with each class. A.T.P.

Synthesis of Carbocyanines. Possible Mechanism of Condensation Reactions

H. Larive and R. Dennilauger

Compt. rend. congr. intern. chim. ind., 31^e, Liège, 1958

(Pub. as *Ind. chim. belge*, Suppl.), 2, 499–502 (Pub. 1959)

Chem. Abs., 54 (10 March 1960) 4612

Synthesis of carbocyanines in phenol as the reaction medium using triethylamine instead of pyridine as the condensing agent has enabled identification of methylene bases and β -alkoxyalkenes whose existence *in situ* could be anticipated but could not hitherto be established. Knowledge of the mechanism peculiar to these reactions has enabled, in some cases, considerable improvement of the yields obtained in such syntheses. C.O.C.

Synthesis of Cyanine Dyes by Cleavage of Chromone and Its Derivatives with Compounds containing Active Methyl Groups

A. I. Kiprianov and A. I. Tolmachev

Compt. rend. congr. intern. chim. ind., 31^e, Liège, 1958

(Pub. as *Ind. chim. belge*, Suppl.), 2, 503–504 (Pub. 1959)

Chem. Abs., 54 (10 March 1960) 6092

Dimethylaniline or *N*-containing heterocyclic compounds having active methylene groups condense with 4-methoxybenzopyrylium salts by replacing the methoxy group. These condensates then react with compounds containing methyl or methylene groups in presence of Na acetate or pyridine to form cyanine dyes by cleavage of the pyrylium nucleus. C.O.C.

Cyanine Dyes. XII—Certain 5,5'-Dimethoxy-6,6'-Diaminothiobarbocyanines

I. I. Levkoev, N. N. Sveshnikov, N. S. Barvyn', and T. V. Krasnova

Zhur. obshch. khim., 30 (Jan 1960) 291–299

Detailed syntheses of symmetrical 5,5'-dimethoxythiobarbocyanines containing free or substituted amino groups in 6,6'-positions are given. Other dyes having 6,6'-*p*-toluene sulphonamide or 6,6'-bis(*N*-methyl-*N*-acylamino) substituents were also synthesised. In each case introduction of the methoxy group had a bathochromic effect, the only exception being 5,5'-dimethoxy-6,6'-bis(dimethylamino)thiobarbocyanine. Acetylation of methylamino groups in a heterocyclic nucleus causes a greater hypsochromic effect and decrease in basicity of the dye than acetylation of amino groups. This is caused by formation of an intramolecular hydrogen bond between the nitrogen and the oxygen of the acetyl group which decreases the electron-attracting properties of the whole substituent. L.S.L.

Reactive Dyes—Constitutions and Synthesis

Bao Chi-min

Zhur. priklad. khim., **33** (March 1960) 700-706

The principles involved in the synthesis of reactive dyes and in their reaction with cellulose are discussed. The dye is said to contain a colour-contributing component which can be an azo, anthraquinone or phthalocyanine derivative and a reactive group—acetyl chloride, cyanuric chloride, isocyanate, vinyl sulphone, ethyleneimine or others. The reaction with cellulose will occur via -OH group either in alkaline medium or at high temperatures. The formulae of most of the commercial dyes and some of the methods of synthesis are also given.

L.S.L.

Progress in Leather Dyes

H. E. Nursten

J. Soc. Leather Trades Chem., **44** (April 1960) 182-191

A discussion of recent progress in making fresh dyes available for leather dyeing, basing its comments largely on the constitutions disclosed in the second edition of the *Colour Index*. Anionic dyes, aftertreatment, metal complex acid dyes, and reactive dyes are particularly considered.

P.B.S.

New Method of Preparing Pigments for Leather Finishes

F. Belohradsky and M. Kulhanek

Veda a Vyzcum v Prumyslu Kozedelnem, **5** (1958) 101-112

J. Amer. Leather Chem. Assoc., **55** (April 1960) 245
Precipitation of organic dyes by amino resins is described. The resin may be prepared from either phenol or *o*-cresol. The resin sulphate is strongly dissociated in solution and reacts with dyes to form insoluble pigments. Suitable substrates for the precipitation are BaSO₄, TiO₂, and coloured inorganic pigments. The resulting pigments have lower sp.gr. than those obtained with inorganic reagents, e.g. phosphomolybdic acid. The substrate is dispersed in a 3-5% solution of the dye and a 50% solution of the resin is then added at 40°C., the dry weights of the dye and the resin being the same. The pigment is filtered off, washed free from chlorides and sulphates, dried, and ground. Pure pigments without a substrate can be obtained by precipitating at a higher temperature. The resin-dye complex may coagulate especially in the case of red, orange, and yellow dyes. Recommendations are given for the best dyes and substrates for each colour and fastness requirements. Interesting black pigments are obtained by precipitating Nigrosine (C.I. Acid Black 2) on Carbon Black (C.I. Pigment Black 6 and 7).

C.O.C.

Chronic Tolerance of Annatto (C.I. Natural Orange 4)

G. J. van Esch, H. van Genderen, and H. H. Vink

Z. Lebensm.-Untersuch. u. Frsch., **111** (1959) 93-108
Chem. Abs., **54** (10 March 1960) 4942

Two commercial Annattos were used for colouring margarine were tested by the "Procedures for the testing of food additives in order to establish their safety for use", *World Health Organisation Tech. Rept. Ser. No. 144* (1958) and shown to be not carcinogenic.

C.O.C.

Vegetable Tannins. III—Divi Divi Plant (C.I. Natural Brown 6)

J. B. Rao, K. N. S. Sastry, and Y. Nayvdamma

Bull. Central Leather Research Inst., Madras, **6** (1959) 59-68

J. Amer. Leather Chem. Assoc., **55** (April 1960) 249
Deals with the composition of extracts from various parts of the plant.

C.O.C.

Myrobalans (C.I. Natural Brown 6)

D. E. Hathway

Tropical Sci., **1** (1959) 85-106
J. Amer. Leather Chem. Assoc., **55** (April 1960) 243

Review, 68 references.

C.O.C.

Photochemical and Spectral Properties of Bacterioviridin of Green Sulphur Bacteria

A. A. Krasnovskii and E. V. Pakshina

Doklady Akad. Nauk S.S.S.R., **127** (1 Aug 1959) 913-916

Properties of bacterioviridin (BA), a green pigment obtained from the culture *Chlorobium Lamicola*, have been investigated spectrophotometrically (absorption and fluorescence spectra) in solid films, colloidal and true solutions of BA, and in cells of live bacteria. Absorption spectrum of BA in pyridine (350-900 mμ.) resembles closely

that of chlorophyll-a (CA); but the two pigments differ in their photochemical properties: in the system pigment-ascorbic acid, there are more products of irreversible reduction with BA than with CA (e.g. Methyl Red undergoes irreversible photoreduction above 700 mμ.); also BA is less stable to photochemical oxidation by oxygen from air than CA. It is suggested that the bulk of BA in photosensitising bacteria is in an aggregated and ordered form. Principal max. of fluorescence spectra of BA, taken at -150°C., are given.

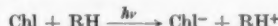
G.J.K.

Mechanism of Photosensitisation of Redox Reactions by Chlorophyll (C.I. 75810) in Solution derived from Conductance Measurements

V. B. Evstigneev and V. A. Gavrilova

Doklady Akad. Nauk S.S.S.R., **127** (1 July 1959) 198-201

Effect of certain dyes (Methyl Red (C.I. 13020), Safranin T (C.I. 50240), Acid Blue (C.I. 42770) and Carotene (C.I. 75130)) on the change in electrolytic conductance of solutions of Chlorophyll-a (Chl) in pyridine in presence of phenylhydrazine (RH) and absence and presence of light, has been investigated. The authors disagree with Livingston's views (*J. Amer. Chem. Soc.*, **77** (1955) 2179) of a metastable triply excited state of the pigment and postulate an intermediate, ionised, photoreduced form of the pigment (Chl⁻) which causes sensitised reduction of the dye or Carotene (Z)—



Z = Safranin T gave the most spectacular changes in conductance. Identical experiments with solutions of pheophytin in pyridine revealed a less characteristic picture than that obtained for Chlorophyll-a. G.J.K.

Photosensitisation of Redox Reactions by Chlorophyll under Heterogeneous Conditions (in Aqueous Suspension)

V. B. Evstigneev and V. A. Gavrilova

Doklady Akad. Nauk S.S.S.R., **126** (11 May 1959) 410-413

Photosensitisation by aq. suspension of Chlorophyll-a and -b (C.I. 75810), pheophytin, or Mg phthalocyanine in presence of ascorbic acid and an azo dye (Methyl Red (C.I. 13020), Acid Blue, or Acid Orange) has been investigated. It is suggested that sensitisation occurs via formation of ion-radicals (I) at the surface of the pigment, the latter behaving on irradiation in presence of an electron donor as a polyradical, thus rendering sensitisation sufficiently effective. This mechanism is extended to granules of chloroplasts. Aq. suspensions of chlorophyll and pheophytin are prepared by pouring small amounts of their conc. soln. in ethanol into excess water, adding 1-2% MgCl₂ or MnCl₂ with vigorous shaking; the residue is filtered, washed with water, and finally suspended in a soln. of ascorbic acid and an azo dye. The mean particle size of the suspended pigment is 10-20 μ. Sensitisation is more effective in acid soln. Presence of air has only slight effect.

G.J.K.

Rafaelite—a High-melting Asphaltum (C.I. Natural Black 6)

H. W. Chatfield

Paint Manuf., **29** (1959) 361-364
Chem. Abs., **54** (10 March 1960) 5121

Freedom from inorganic material, low moisture absorption, high m.p. and limited solubility in true solvents for nitrocellulose, make this product suitable for use where a dark colour is not objectionable.

C.O.C.

Some Relations between the Chemical Constitution of Cibacron Dyes and their Dyeing Characteristics

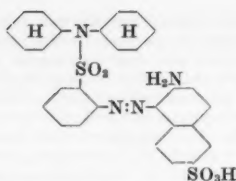
J. Wegmann

J.S.D.C., **76** (April 1960) 205-209**PATENTS****Orange and Red Monoazo Acid Dyes**

Gy

BP 831,843 (Switzerland 5 Dec 1956)

Neutral-dyeing acid dyes are made by diazotising derivatives of orthonilamide containing 2 saturated organic substituents on the N atom of the sulphonamide group which together have > 11 C atoms of which > 9 form part of alicyclic radicals, and coupling with *o*-coupling naphthylamines. Thus diazotised orthanildicyclohexylamide is acid-coupled with 2-naphthylamine-6-sulphonic acid giving—

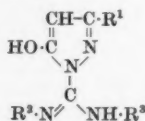


which dyes wool orange from a bath containing $(\text{NH}_4)_2\text{SO}_4$.
E.S.

Yellow Basic Monoazo Dyes for Polyacrylonitrile, etc., from 1-Amidinopyrazolones

FBy BP 832,285 (Germany 13 Oct 1956)

1-Amidinopyrazolones—

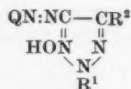


($\text{R}^1 = \text{H}$, Alk, Ar, a carboxylic acid ester or alkylcarboxylic acid ester group; $\text{R}^2, \text{R}^3 = \text{H}$, Alk, aralkyl, Ar, or alkylene groups linked together to form a ring) are coupled with diazo compounds free from SO_3H or COOH groups to give the title dyes. Thus diazotised aniline is coupled with 1-amidino-3-methyl-5-pyrazolone at pH 4–6. The product in the form of its hydrochloride dyes polyacrylonitrile fibres greenish-yellow.
E.S.

Metallisable Monoazo Pyrazolone Disperse Dyes

Eastman Kodak Co. BP 832,199 (U.S.A. 30 Mar 1955)

Monoazo compounds—

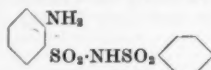


($\text{Q} =$ benzene nucleus containing 1 or 2 groups OCH_3 , OC_2H_5 , or $\text{OC}_2\text{H}_4\text{OH}$, either a single group *o*- or *p*- to the azo group, or 2 groups in the 2- and 5-positions; $\text{R}^1 = \text{H}$, phenyl, or subst. phenyl; $\text{R}^2 = \text{CH}_3$, $\text{CO}-\text{OCH}_3$, $\text{CO}-\text{OC}_2\text{H}_5$, NH_2 , OH , or CF_3) free of COOH and SO_3H groups may be metallised, especially with Ni compounds, on cellulose acetate, or the metal complexes formed in substance may be used for dyeing nylon and acrylonitrile polymers, etc., or incorporated in the spinning solutions of e.g. cellulose acetate. The metal complexes contain 2 monoazo dye mols. to 1 metal atom, and the alkyl radicals are not split off from the OAlk groups by metallising. Thus 2,5-dimethoxyaniline is diazotised and coupled with 3-methyl-1-phenyl-5-pyrazolone. The monoazo compound so formed dyes cellulose acetate bright yellow, whose fastness to light and wet treatments is much increased by padding with aq. $\text{Ni}(\text{SCN})_2$ and steaming.
E.S.

Monoazo Pyrazolone Acid Dyes having Sulphon-(sulphonyl)amide Groups

FBy BP 831,840 (Germany 5 Dec 1956)

Arylamines $\text{R}^1\text{SO}_2\text{NH}\cdot\text{SO}_2\text{R}^2\text{NH}_2$ ($\text{R}^1 = \text{Alk}$ or Ar of benzene series free of SO_3H or COOH groups; $\text{R}^2 = \text{Ar}$ of benzene series free of SO_3H or COOH groups) are diazotised and coupled with 1-sulphoaryl-3-methyl-5-pyrazolones or 1-sulphoaryl-3-methyl-5-aminopyrazolones to give yellow acid dyes applicable from weakly acid baths. Thus aniline-2-sulphon(phenylsulphonyl)amide—



is diazotised and coupled with 1-*p*-sulphophenyl-3-methyl-5-pyrazolone.
E.S.

Spirit-soluble Monoazo Compound—Basic Dye—Metal Complexes

Ciba BP 832,206 (Switzerland 21 April 1955)

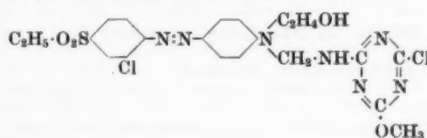
Metal-complexes containing 1 atom of Co or Cr to 2 mol. of *oo'*-dihydroxy- or *o*-amino-*o'*-hydroxymonoazo compounds free of SO_3H and COOH groups, but containing a sulphone or sulphamyl group, are combined with basic

dyes to give spirit-soluble colorants for lacquers, fibre spinning solutions, etc. Thus the monoazo compound 2-aminophenol-4-sulphonamide→acetoacetanilide is stirred with aq. CoSO_4 to give the Co-complex. A solution of Rhodamine B (C.I. Basic Violet 10) is then added. The complex so formed colours cellulose nitrate lacquer yellowish red.
E.S.

Monoazo Disperse Dyes for Nylon having a Triazine Ring

ICI BP 833,396 (15 June 1956)

Monoazo compounds free from water-solubilising groups and containing a monohalogenotriazinylamino residue attached to the residue of a monoazo compound containing a primary or secondary amino group through an alkylene chain, dye nylon yellow to red of very good wet fastness, and may also be used on cellulose acetates. Thus 2,4-dichloro-6-methoxy-1,3,5-triazine is condensed in acetone with 1 mol. of *N*-β-aminoethyl-*N*-β-hydroxy-ethylamine in presence of NaHCO_3 , and the product is coupled with 1 mol. of diazotised 2-chloro-4-ethylsulphonylaniline to give—



which dyes nylon orange.

E.S.

Brown and Black Azoic Dyes for Nylon, etc.

FBy BP 833,669 (Germany 2 July 1955)

Tetrazotised benzidine, or its derivatives containing Hal, Alk, or OAlk substituents, couples on nylon and polyurethane fibres with arylides of 5,6,7,8-tetrahydro-3-hydroxy-2-naphthoic acid (I) to give fast browns and blacks; the benzidide (or subst. benzidide) of I gives dyeings and prints of especially high resistance to steaming at high temperatures. Thus nylon yarn is worked at 85°C. in a bath containing the *o*-dianiside of I dissolved in alcoholic NaOH , and the bis-triazene from tetrazotised benzidine condensed with 2 mol. of diethanolamine. After treatment in an acid bath develops the yellow brown azoic dye.
E.S.

Anthraquinone Disperse Dyes

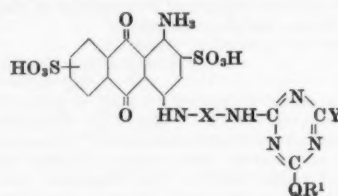
S BP 833,132 (Switzerland 15 Feb 1957)

Synthetic fibres are dyed brilliant blue of excellent fastness to light, washing, perspiration, sublimation, and gas-fumes by the condensates of 1 mol. of 1,4-diaminoanthraquinone-2,3-dicarboxylic acid (I) with at least 2 mol. of a sulphonc acid ester, $\text{R}^2\text{SO}_2\text{OR}^1$ ($\text{R}^1 = \text{subst.}$ or unsubst. Alk, and $\text{R}^2 =$ aromatic, aliphatic, cycloaliphatic or aryl-aliphatic). Condensation is effected at 20–120°C. (preferably 70–95°C.) in a weakly basic organic medium or in dimethyl sulphoxide and in presence of an acid binding agent and absence of water. Thus I (8), chlorobenzene (40), pyridine (4), NaHCO_3 (10), and benzene sulphonc acid ethyl ester (23) are stirred at 86°C. for 16 hr. and the liquor run into water (200). The mixture is weakly acidified (HCl) and chlorobenzene distilled in steam. The blue precipitate is collected and purified by dissolving in pyridine, filtering, and precipitating with NaOH . It dyes sec. acetate a clear blue of excellent fastness.
A.T.P.

Anthraquinone Dyes

ICI BP 832,400 (22 July 1957)

Greenish-blue to green dyes for cotton and other fibres and having excellent solubility and fastness to light and washing have formula—

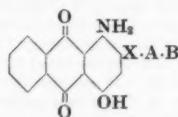


(X = bridging group containing < 1 anionic solubilising group; Y = Cl or Br; Q = O, S or NR² (R² = H or subst. or unsubst. Alk); R¹ = H or subst. or unsubst. hydrocarbon). They have greater stability in presence of acid binding agents than the similar dyes of BP 781,930 (J.S.D.C., 73 (1957) 524; where Y and QR¹ are both replaced by Cl), thus facilitating their application, e.g. in thickened printing pastes containing the dye and an acid-binding agent. Thus, greenish-blue dyes are obtained by reacting substantially equimolecular proportions of 1-amino-4-(4'-aminoanilino)-anthraquinone-2,3',5'-trisulphonic acid (I), cyanuric chloride and, e.g. phenol, *o*-cresol, 3-sulphoaniline or thio-*p*-cresol. A similar dye is that from I and 2,4-dichloro-6-methoxy-*s*-triazine. A.T.P.

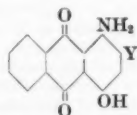
1-Amino-2-alkoxy- and 2-alkylthio-4-hydroxy-anthraquinones—Red Disperse Dyes

Ciba BP 834,949 (Switzerland 17 Oct 1957)

Compounds of formula—



(X = O or S; A = alkylene, especially methylene; B = Ar, especially benzene) are bluish-red dyes of excellent light and sublimation fastness on synthetic fibres, especially Terylene. They have greater brilliance and colouring power than previously known 2-oxoalkoxy subst. dyes and are obtained by condensation of Z.A.B. with—



(A, B = as above; one of Z and Y = Hal or aryloxy, the other OH or mercapto). Thus, 1-amino-2-phenoxy-4-hydroxyanthraquinone (I) (9), benzyl alcohol (90), and KOH (2) are heated at 120–125°C., until no further I is present, to give a bright yellowish-red dye of excellent fastness on Terylene. Similarly 1-amino-2-bromo-4-hydroxyanthraquinone (100) is heated with 60% Na₂S (130), S (50), H₂O (240) and C₂H₅OH (300) for 5 hr. at 90°C. in an autoclave. The solution is made up to volume (2000) with water and the 1-amino-2-mercapto-4-hydroxyanthraquinone, precipitated at 0°C. to –5°C. by NaCl (400), is then dissolved in C₂H₅OH (1000) and benzyl chloride added at 75°C. until the bluish violet colour disappears. The product dyes Terylene a brilliant bluish-red of excellent fastness. A.T.P.

Halogenated Amino-Hydroxyanthraquinones—Blue Disperse Dyes

By BP 834,518 (Germany 27 Sept 1957)

Halogenated derivatives of 5,8-diamino-1,4-dihydroxyanthraquinone, 4,5,8-triamino-1-hydroxyanthraquinone, and 8-amino-1,4,5-trihydroxyanthraquinone (I) are blue disperse dyes of good sublimation and light fastness. 0.5–1.5 Halogen atoms per anthraquinone molecule are advantageous for maximum affinity and fastness. Thus I (27.1) and H₃BO₃ (13.5) are dissolved in monohydrate (270) and I₂ (0.1) and Br₂ (20) added. The mixture is heated at 50°C. until the product contains 20% Br. Drowning into ice gives a strong reddish-blue dye of excellent properties on Terylene. A.T.P.

Anthraquinone Disperse Dyes

ICI BP 833,832 (24 Jan 1958)

Red, purple, and blue dyes of excellent washing fastness on nylon contain at least one γ -bromo- β -hydroxypropyl-amino- or di-(γ -bromo- β -hydroxypropyl)aminoaryl-amino group. They are obtained by condensing amino- or aminoaryl-amino-anthraquinones with epibromohydrin (I). Thus, I (70) is added to a stirred suspension of 1-amino-4- β -hydroxyethylaminoanthraquinone (254) in acetic acid (130) and H₂O (25) at 20°C. The mixture is stirred at 80°C. and further I (176.6) added over 3 hr., the temperature then being maintained at 80°C. for 4 hr. Cooling (20°C.) and precipitation by acetic acid (45) and H₂O (250) gives a

reddish-blue dye of excellent washing fastness on nylon. The dyes are much faster to washing than similar dyes from epichlorohydrin and may also be used on other synthetic fibres, but it is preferable here to treat the fibre either before, during or after dyeing with an amine, phenol or thiophenol. A.T.P.

Iso-Indolone Dyes and Pigments and Their Use

Gy BP 833,548 (Switzerland 13 July 1956)

Coloured condensation products are obtained from 1 mol. of a polyprimary amino compound of the homo- or heterocyclic aromatic series or hydrazine and 1 mol. equivalent for every primary NH₂ group of the same or different iso-indole-1-one, subst. by < 3 Hal in the benzo radical, in the 3-position by an imino group which may be organically subst., a doubly bound S atom, two Hal atoms, two *s*-amino groups or two ether groups. The water-insoluble products have excellent light fastness, high colour strength and fastness to migration in plastics and are valuable pigments for, e.g. rubber, lacquers, and in finely dispersed form can be used for colouring paper, viscose, cellulose esters or polyamides in the spinning mass. Products containing water-solubilising salt-forming groups yield insoluble lakes with Ca, Ba, Sr, Al or heavy metal salts, or, depending on their affinity, can be used for dyeing paper and cellulosic or animal fibres. Thus, a solution of 1,4-diaminobenzene (5.4) in *o*-dichlorobenzene (I) (200) is added to a solution of 3,3,4,5,6,7-hexachloro-isoindole-1-one (34) (prepared from PCl₅ and tetrachlorophthalimide) in I (300). The yellow suspension is heated at 160–170°C. for 3 hr., filtered hot and washed with I, ethanol and acetone to give an orange-yellow pigment (32) of excellent fastness to light, migration, and solvents. A.T.P.

Acylated Leuco Methylene Blue

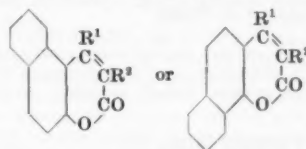
General Aniline BP 834,039 (U.S.A. 8 Nov 1955)

Acylated Leuco Methylene Blue is obtained in excellent yield and high purity by acylation of Leuco Methylene Blue with an aromatic acylating agent, especially benzoyl chloride, in aq. medium in presence of a water-immiscible organic solvent for the acylated product, e.g. chlorobenzene, chloroform, at pH 3–6 (preferably 4) and preferably in presence of a metal sequestrant. The products are used in "carbonless carbon papers" and the high degree of purity required for this is satisfied in the present process. Thus, a mixture of distilled water (300 ml.), chlorobenzene (250 ml.), Methylene Blue Chloride (32) (C.I. Basic Blue 9), and ethylene diamine tetraacetic acid (5) is reduced with Na hydrosulphite (5) and 15 N. NaOH (13.3). At pH 6.5, benzoyl chloride (35 ml.) is added and pH allowed to fall to 4, and maintained at 4 over 2 hr. by NaOH additions. Decolourising charcoal (5) and attapulgus clay (7) are added and after addition of further NaOH to pH 10, the mass is filtered. The organic layer is separated and distilled to dryness in vacuo to give crystalline benzoyl-Leuco Methylene Blue (36) of high purity. A.T.P.

Fluorescent Brightening Agents

FBy BP 834,903 (Germany 28 Sept 1955)

Colourless or nearly colourless fluorescent benzocoumarin compounds, particularly those of formula—

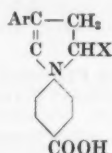


(R¹ = H or Alk; R² = H, Alk, Ar, acyl, carboxyl (which may be esterified), carbonamide (which may be substituted by Alk or aralkyl) or cyano; the aromatic rings as well as the aromatic ring possibly contained in R³ may carry one or more substituents) when soluble in water are good fluorescent brightening agents for animal or vegetable fibres and when insoluble in water are very suitable for brightening synthetic polycondensates, polyacrylonitriles, and cellulose esters. Thus 5,6-benzocoumarin-3-carboxylic acid in aqueous dispersion gives excellent brightening of polyacrylonitrile and polyester fibres and the Na salt of sulphonated 5,6-benzocoumarin-3-carboxylic ethyl ester has good affinity from aqueous solution for wool. C.O.C.

Arylpyrazolines as Fluorescent Brightening Agents

FBY BP 832,239 (Germany 10 Oct 1955)

Compounds of formula—



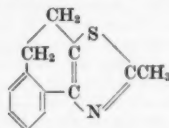
(X = H or Ar; the aromatic nuclei may be further substituted by Hal, OH, Alk of 1-5 C or alkoxy), e.g. 1-*p*-carboxyphenyl-3-phenylpyrazoline, are good brightening agents for polyacrylonitrile products, soaps, and detergents.

C.O.C.

Cyanine Dyes from 2-Methyl-4,5-dihydro-β-naphthothiazole

Sperry Rand Corp. USP 2,905,666 (10 May 1955)

2-Methyl-4,5-dihydro-β-naphthothiazole—



is converted into a quaternary salt by treating it with an ester, e.g. alkylhalide, alkylsulphate or alkyl-*p*-toluene sulphate. These salts are then treated in known manner to yield cyanine dyes in which one or both of the auxochromic N atoms is in a 4,5-dihydro-β-naphthothiazole nucleus.

C.O.C.

Calcium Silicate and Calcium Silicate-Silicic Acid BASF

BP 833,607 (Germany 18 Dec 1956)

A product having an especially large surface area is obtained by treating a silicic acid sol. or a suspension of a silicic acid hydrogel of particle size $> 0.3 \mu$ with $\text{Ca}(\text{OH})_2$, drying and grinding. It consists of fine lamellae of thickness 5μ , and length and breadth 50–200 μ . It is used as a filler for rubber and plastics.

C.O.C.

Calcium Sulphate-Aluminium Silicate Pigment

R. T. Vanderbilt Co. BP 832,636 (2 March 1956)

Pigments suitable for use in the manufacture of paper, rubber, paints, and ceramics are obtained by vigorously agitating together in absence of cellulosic fibres aqueous solutions of a Ca salt and a silicate. The resulting finely divided hydrated Ca silicate is treated without being dried with Al sulphate ($< 1\% \text{ Al}_2\text{O}_3$ on wt. of Ca silicate).

C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7)

Columbian Carbon Co. BP 833,411 (U.S.A. 16 July 1957)

A new method for injecting the hydrocarbon make into the hot gas stream when preparing Carbon Black by the process described in BP 652,518 (J.S.D.C., 67 (1951) 319).

C.O.C.

Chromic Oxide (C.I. 77288)

F. L. Melvill BP 833,563 (26 Feb 1957)

Chromic oxide of high purity is economically prepared by treating a chromate and/or dichromate with Al_2O_3 and/or Al phosphate under reducing conditions at $< 1000^\circ\text{F}$.

C.O.C.

Silica (C.I. Pigment White 27)

Columbia-Southern Chemical Corp.

USP 2,905,567 (18 Oct 1956)

A finely divided, precipitated, hydrated, porous, siliceous floc containing $> 90\% \text{ SiO}_2$ (on wt. of floc excluding bound and free water) and 1 mole of Al and/or Fe to 40–350 moles of SiO_2 is a good and inexpensive reinforcing pigment for rubber. Presence of the Fe and/or Al results in the pigment separating rapidly when it is precipitated during its manufacture.

C.O.C.

Hydrophilic Fluorine-free Silicon Dioxide (C.I. Pigment White 27)

G. L. Flemmert BP 833,143 (Sweden 25 March 1957)

When hydrophobic SiO_2 is treated with water vapour at $> 500^\circ\text{C}$. until the neutralisation number is < 15 , it

becomes strongly hydrophilic but has no tendency to agglomerate on storage in air.

C.O.C.

Dyeing Mechanisms and Molecular Shape (VIII p. 460)
o-Dihydricphenols as Oxidation Dyes for Hair (VIII p. 461)

V—PAINTS; ENAMELS; INKS

Lead Orthoplumbate in Ground Coatings

J. Jafusek and M. Svoboda

Korose a ochrana materiálu, 3 (1959) 745

Chem. Abs., 54 (10 March 1960) 5119

Laboratory trials show that the anticorrosive properties of ground coats pigmented with $2\text{CaO} \cdot \text{PbO}_2$ or Pb_2O_3 are similar.

C.O.C.

Effect of Orientation in Lacquer Films containing Carbon Black as Filler

B. S. Gal'perin and L. P. Soldatova

Kolloid. zhur., 21 (July-Aug 1959) 415–418

Films (5–7 μ . thick) have been obtained by dipping porcelain rods into lacquer containing 8–15% Carbon Black (C.I. Pigment Black 6 & 7), allowing to drain, and drying in air. Measurements of specific conductance of the film in the direction of drain and perpendicular to it, at varying volume concn. of pigment, revealed anisotropy of conductance (AC) which is attributed to preferential orientation of Carbon Black chains in the direction of drain of suspension. AC can be eliminated by adding 2–3% plasticiser or by preparing films from fatty (relatively slow drying) lacquers.

G.J.K.

PATENTS

Transfer Coating Compositions

Kolok Manufacturing Co. BP 833,631 (12 Aug 1955)

A solvent dye is dispersed in a non-solvent volatile organic liquid which evaporates at room temperature. This liquid has dissolved or dispersed in it a thermoplastic synthetic resin and/or a cellulose ether and/or chlorinated rubber. The preparation of components are such that when the composition is applied to a base sheet and dried the resulting transfer coating contains $< 25\%$ by weight of dye.

C.O.C.

Reflective Pigments and Coating Compositions

Minnesota Mining & Manufacturing Co.

BP 833,651 (U.S.A. 25 April 1955)

Transparent glass microspheroids of flour-like fineness are given a hemispherical reflective coating of metal and then used to pigment transparent varnishes and lacquers. The resulting reflective varnishes can be applied by spraying, painting, printing, and screen-processing.

C.O.C.

Mixture of a Thermoplastic Copolymer and a Thermosetting Aminoplast Resin as Vehicle for Enamels, Printing Inks, and Stoving Enamels

Rohm & Haas Co. BP 831,898 (U.S.A. 27 July 1956)

A mixture of (a) 5–95 parts by wt. of a linear, water-insoluble, thermoplastic copolymer of acrylic, methacrylic or itaconic acid (1–10 mole %) and an alkylacrylate or methacrylate (90–99) and (b) 95–5 parts of a thermosetting alkoxyethyl aminoplast condensate (alkoxyethyl of 2–19 C) on baking yields a hard, insoluble clear film having good gloss, colour, and resistance to heat and chemicals.

C.O.C.

Coating of Metal

DuP

BP 833,122 (U.S.A. 17 Jan 1957)

A pigmented primer coating for use under either methyl methacrylate or nitrocellulose lacquers consists of a stoved layer of a coating composition comprising an alkyl resin formed from (a) dicarboxylic acids of which < 25 mole % are hexachloroendomethylenetetrahydrophthalic acid, (b) tall, semi-drying or drying oil or a fatty acid derivative from any of these oils, and (c) a polyol. The alkyl resin has an oil length of 20–45, acid number < 50 and unreacted hydroxyl equivalent to 0.5–5.0% by wt. of glycerol on the total wt. of alkyl resin.

C.O.C.

Aluminium Alcoholate Derivatives for Increasing the Consistency of Paints, Varnish, and Lacquer Media

Hardman & Holden

BP 832,622 (5 March 1956)

Addition of an alkoxyaluminium acylate, dialkoxy aluminium acylate, monoalkoxy aluminium diacylate, Al triacylate, Al alcoholate or acylate in which one or more alkoxy or acylate groups have been replaced by

aryloxy groups and polymeric aluminium-containing derivatives, e.g. those described in BP 806,113 and 809,309, to paint media based on oils and resins followed by their mutual reaction at ordinary or raised temperature results in increased consistency of the media. C.O.C.

Aqueous Dispersion Coating Compositions

DuP BP 832,111 (U.S.A. 11 Oct 1956)

An aqueous dispersion of a copolymer of a vinylbenzene monomer (20-70 parts) an α -olefinic monocarboxylic acid having a methylene group doubly-bonded to the α -C atom (1-20) and an ester of such an acid with a std. aliphatic alcohol of 1-8 C (to bring to 100) has all the advantages of an aqueous dispersion coating agent and, in addition, is a good primer coating. C.O.C.

Chemically-bonded Coatings on Aluminium

Amchem Products BP 832,485 (U.S.A. 4 Feb 1957)

A bath is used containing fluoride ion (0.15-12.5 g./litre), hexavalent Cr (3.0-60.0 CrO₃), and phosphate (2.0-285.0 PO₄). The bath is at pH 0.8-4.5 and also contains Na and K (0.0026-0.0526 mol. total alkali metal per g. of fluoride ion). The amount of dissolved Al is governed by the solubility limit of NaK fluoaluminate containing < 6 F atoms per Al atom. This yields coatings of constant colour and uncontaminated by powder.

BP 832,486 (U.S.A. 4 Feb 1957)

Replenishment material for the above bath comprises sources of F, Na, and K, there being 0.0065-0.0455 mol. total alkali metal per g. F, e.g. a mixture of NaHF (44.2 g.) and KHF (55.8 g.). C.O.C.

VI—FIBRES; YARNS; FABRICS

Sulphur Balance of Wool

G. Wibaux, G. Mazingue, and M. van Overbèke

Bull. Inst. Text. France (86) (Jan-Feb 1960) 41-53

Estimations of total S in wool, wool-cystine mixtures, and wool-lanthionine mixtures by methods of Parr bomb, Benedict-Denis, and Kahane (Osterr. Chem.-Zeit., 55, 15 (1954)) gave concordant results. The total S figure is thus reliable. The amounts of S present as (a) cystine and cysteine (Shimohara), (b) methionine, (c) lanthionine, (d) cysteic acid, (e) total H₂SO₄, (f) free H₂SO₄, (g) Br-oxidisable S were determined in (i) untreated wool, (ii) acid-treated wool, (iii) alkali-treated wool, (iv) carbonised wool with and without neutralisation. The sum of the various S contributions, excluding (e) and (g), agreed with the total S for (i) and (ii), and for (iv) if (e) was substituted for (f) to take account of combined H₂SO₄. For (iii), 13% of the total S remained unaccounted for. It is concluded that S-balance can be achieved for wool provided significant breakdown of cystine has not occurred. The importance of taking account of the small amounts of cysteine, lanthionine, and cysteic acid in untreated wool is emphasised. J.C.F.

Mechano-chemical Modifications of Wool Caused by Alkali Treatment

G. Satlow Melliand Textilber., 40 (Dec 1959) 1426-1429

The stress-strain curves of wool fibres treated under mild conditions with alkali were compared with the curves for untreated wool. It was found that the curves are not parallel, i.e. the effect of the treatment is not the same in different parts of the curves. In the region of greater loads higher values are required to get the same extension with treated than with untreated samples. The hypothesis is suggested that this is caused by the cross-linking effect of the alkali treatment. W.M.

The Chemical Modification of Wool by Trichloroacrylodiazomethane and 1,4-Dichlorobutene-2

A. Kling Melliand Textilber., 41 (April 1960) 457-462

A bifunctional compound is used having a triple carbon-carbon bond for the cross-linking of wool. Presence of the triple bond prevents both end-groups reacting with one and the same chain. Chemical and mechanical tests showed that cross-linkage had actually taken place. Efforts to identify the sites in the keratin where the bridging occurred were unsuccessful. W.M.

Orlon Cantreze—Du Pont's Newest Sweater Fibre

H. T. Pratt Amer. Dyestuff Rep., 49 (6) (21 March 1960) 41-43 (190-192)

Recommended methods for texturing, package, and piece dyeing and the finishing of garments made from package-dyed yarns. C.O.C.

Study on the Oligo-amides of Rilsan

P. Miró

Investigación e Información Textil,

2 (Jan-March 1959) 13-18

An oligo-amide of m.p. 188-189°C. corresponding to a cyclic dimer of ω -amino undecanoic acid, can be isolated from Rilsan together with an oligo-amide of m.p. 159-160°C. corresponding to a cyclic trimer of the same acid. Linear oligo-amides can also be detected. S.R.C.

Effect of Sulphuric Acid on Wool. IV—Some Further Observations of the Reaction with Concentrated Acid

R. L. Elliott, R. S. Asquith, M. E. P. Hopper, and D. H. Rawson

J.S.D.C., 76 (April 1960) 222-225

PATENT

Conditioning Method for Improving the Affinity for Dyes of Stretched Wet-spun Polyacrylic Fibres

Dow Chemical Co. BP 830,986 (U.S.A. 27 Aug 1956)

The fibres while in aqagel form are steamed at 20 lb./sq.in. for 0.5 sec.-0.5 hr. and then dried. The treated fibres yield dyeings of excellent penetration with a wide variety of dyes applied by conventional methods. C.O.C.

Polymeric Amidoximes: Auxiliary Agents: Increasing the

Affinity of Acrylonitrile Fibres for Dyes (VIII p. 462)

Chemically-reactive Allylcellulose (XI p. 465)

States of Aggregation of High-molecular Compounds.

IV—Collagen, Gelatine, and Silk Fibroin (XII p. 466)

β -Resorcylic Acid Diesters for Stabilising Hydrocarbon

Polymers to Ultraviolet Radiation (XIII p. 467)

Stabilising Halogenoethylene Polymers to Light (XIII

p. 467)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Origins of Chlorine Bleaching in America

S. M. Edelstein

Amer. Dyestuff Rep., 49 (18 April 1960) 254-263

An illustrated account of the development of chlorine bleaching in the U.S.A. As early as 1798 use of chlorine for bleaching papermaker's rags and paper was the subject of a U.S. patent and chlorine was being manufactured and used for bleaching rags in Delaware by the summer of 1804. A full account of this development is given and the papers discussed also describe the use of chlorine bleaching in several places in England. The site of the works in Delaware where this development took place is now occupied by the works of Joseph Bancroft & Sons Co. However the use of chlorine for bleaching textiles was not introduced into the U.S. until 1830. C.O.C.

Bleaching of Fabrics of Low-grade Cotton

N. I. Birger Tekhnol. tekstil. prom., No. 1 (8) (1959) 182

Removal of mechanical occlusions is facilitated by soaking low-grade cotton fabrics in a soln. containing, per litre, 5 g. Na₂PO₄, 5 g. NaHCO₃, and 0.5 g. OP-10; squeezing, and leaving for 4 hr. before washing for kier boiling and bleaching. G.J.K.

Bleaching Problems in the Textile Industry

B. K. Easton

Canadian Textile J., 77 (18 March 1960) 39-42

Actual cases where problems in bleaching have been traced to faults in grey goods are cited. Pinholes in fabrics and pinpoint breaks in yarn were caused by iron or copper present in the grey material and the sources of the metal (in particular pretreating machinery) were traced. Iron was successfully removed by acid pretreatment followed by an alkaline scour or by incorporating triethanolamine in an alkaline scour. Distortion of the fabric may be caused by maladjustment of the machinery. A cold peroxide bleach sequence is described; a better bleach results if there is no rinse between peroxide bleach and alkali boil. Two examples of the use of dyeing steamers are given, both involve saturating the desized and prepared cloth with alkaline hydrogen peroxide and steaming for 60-90 sec. Complete mote removal, good absorbency and bleach are claimed. Hydrogen peroxide solution (containing Na₂CO₃), padded on to the fabric followed by wet steaming at 165-208°F. eliminates the amine odour, from damp wash-and-wear fabrics, more efficiently than sodium perborate. Yellowing during the

curing of resin finishes is prevented by adding 0.4–0.5% H_2O_2 to the resin mix. It has no adverse effect on wash-and-wear, strength or chlorine resistance. It is ineffective with triazone type finishes. W.P.M.

The Effect of Sodium Chlorite on Bast Fibres

M. Lüttke *Melliand Textilber.*, 41 (April 1960) 437–442
Sodium chlorite does not remove all the lignin but only one component, lignin a. On the other hand carbohydrates are attacked. This loss is more significant with fibres of a low lignin content. Nearly all protein substances present in bast fibres go into solution. Repeated treatment causes disintegration into single fibres more readily with materials with a high lignin content. The substances removed during retting are only partly identical with those removed by chlorite. The lignin left in the holocellulose can be detected by alcoholic phloroglucinol or HCl gas. Complete separation of lignin and carbohydrates by chlorite, chlorine dioxide, or chlorine is not possible. W.M.

Behaviour of Synthetic Polymer Fibres in Carbonising

G. von Hornuff

Melliand Textilber., 41 (April 1960) 474–478
Synthetic polymer fibres in general show good resistance to acids. Polyvinyl-fibres and especially poly(vinyl-chloride) are distinguished by a very high resistance. Polyacrylonitrile fibres are not equally resistant but are superior in this respect to wool. Polyamide fibres are more sensitive to attack by acids than the other synthetic polymer fibres but are sufficiently resistant to the strengths of acid used in dyeing and finishing. The effect of temperature and concentration is discussed. Three methods of carbonising were tried: (1) sulphuric acid, (2) aluminium-chloride, (3) HCl gas. Poly(vinylchloride) fibres are not damaged by acid at 110°C. for 15 min. nor by carbonising but they shrink during the process. Polyacrylonitrile fibres (Wolerylon II) when carbonised with HCl gas suffer very little damage, practically none with $AlCl_3$, and with sulphuric acid under both normal conditions of carbonising and more severe conditions there was no significant loss of tensile strength. Polyamide fibres treated with HCl gas form a plastic mass and the mol.wt. is much reduced. They are resistant to carbonising with $AlCl_3$ or H_2SO_4 , neither the tensile strength nor viscosity showing any significant reduction. W.M.

Action on Cotton Cloth of repeated Bleaching Treatments based on Per-salts

A. Parisot

Bull. Inst. Text. France (86) (Jan–Feb 1960) 127–136
Changes in the mechanical properties of a cotton fabric subjected to repeated bleaching treatments based on sodium perborate were found to obey the same laws previously found to describe the behaviour of the same fabric in bleaching with sodium hypochlorite. The change in mechanical properties could be correlated with the D.P. of the cellulose, and, whatever the conditions of bleaching, the wet and dry strengths of the fabric became equal at a D.P. of about 600. J.C.F.

Investigation of the Carbonising of Wool

H. Zahn *J.S.D.C.*, 76 (April 1960) 226–231

Effect of Sulphuric Acid on Wool. IV—Some Further Observations of the Reaction with Concentrated Acid (VI p. 459)

VIII—DYEING

Dyeing Mechanisms and Molecular Shape

H. Zollinger *Amer. Dyestuff Rep.*, 49 (7 March 1960)

(142)–(149) 29–36
English translation of article first published in *Textil-Rundschau*, 14 (March 1959) 113–125 (*J.S.D.C.*, 75 (July 1959) 402). C.O.C.

Interaction between Dyes and Surfactants—I

T. Kondo and K. Meguro

Bull. Chem. Soc. Japan, 32 (1959) 267–271
The interaction between anionic surfactants with alkyl chains of different lengths and a cationic dye, and between cationic surfactants having gegenions of different degrees of hydration and an anionic dye was studied.

Rhodamine 6G (C.I. Basic Red I) and Eosine (C.I. Acid Red 87) were used as cationic and anionic dyes respectively with decyl, dodecyl, and tetradecyl sulphates as anionic surfactants and dodecylpyridinium chloride, bromide, and iodide as cationic surfactants. Two ml. of dye solution of constant strength and an equal volume of surfactant solution of various strengths were mixed in small test tubes, stood for 24 hr. at 30°C. and the degree of flocculation observed visually or spectrophotometrically, if necessary, after centrifuging. The flocculation and deflocculation values recorded correspond to the minimum number of millimoles of surfactant/l. of mixed solution required to cause complete flocculation and deflocculation. The same procedure for dye solutions of various strengths gave the dependence of flocculation and deflocculation values on the concentration of the dye. Dye-surfactant complexes were studied on flocculate in nonpolar solvent (C_6H_6) the degree of colouring of the C_6H_6 phase being measured photometrically. Flocculation and deflocculation values varied with difference in alkyl chain length, gegenion of surfactant and presence of salt or organic solvent. Minimum amounts of the sulphates required for complete flocculation and deflocculation decreased with increase in alkyl chain length of the sulphates. As the dye concentration decreased the flocculation and deflocculation values decreased. Of the dodecylpyridinium halides, the chloride gave the largest flocculation and deflocculation values and the iodide the smallest. Salt depressed flocculation but promoted dispersion of flocculants. Ethanol in a Rhodamine 6G–Na dodecyl sulphate system did not alter the flocculation value of dodecylsulphate but exerted a slight influence on its deflocculation zone. The Rhodamine 6G–dodecyl sulphate complex in C_6H_6 shows strong oleophilic property when the concentration of the Na dodecyl sulphate is equivalent to that of the dye. This oleophilic property decreases again in stronger solutions of Na dodecyl sulphate. Probably the deflocculation phenomenon of the dye is closely related to the associated properties of surfactant, and thus exists as dye-surfactant complex, as the nucleus, and few surfactant ions. With few surfactant ions combined with a dye-surfactant complex in the deflocculation, the surfactant ions themselves can form an aggregate because of the interaction between their alkyl chains below the critical concentration for micelle formation. C.O.C.

Levelling of Vat Dyes

J. M. Jubany Socas *Investigación e Información Textil*, 2 (July–Sept 1959) 201–213

A discussion of levelling methods, including temperature control, levelling agents, sulphoxylate-formaldehyde, pigment padding, vat acid, solvents, and high temperature. S.R.C.

Thermodynamics of the Vat Dyeing of Cellulose

P. V. Morýganov and M. I. Artým

Tekhnol. tekstil. prom., No. 2 (9) (1959) 125–133
The dependence of the affinity of two polycyclic dyes and one indigoid dye for cuprammonium rayon upon the concn. of surface active agents and organic solvents in the dyebath has been investigated using the thermodynamic treatment proposed by Marshall and Peters (*J.S.D.C.*, 63 (1947) 446). The affinity of these dyes decreases in proportion to increase in concn. of the above agents; the series in decreasing order of effectiveness being: polyvinylpyrrolidone > OP-10 > Cellosolve > pyridine > triethanolamine. G.J.K.

Chemistry of Dyeing. II—Behaviour of Remazol Dyes when Dyed by the Exhaustion Process

H.-U. von der Eltz and F. Osterloh

Melliand Textilber., 40 (Dec 1959) 1443–1448

The chemical mechanism of the fixation of Remazol dyes on cellulosic fibres is briefly explained. The authors then discuss in some detail how the result of dyeing is affected by the following factors: temperature, pH, time of dyeing, concentration of electrolyte, type of electrolyte, liquor length, and type of cellulosic fibre. The authors state that the amount of dye taken up under otherwise identical conditions increases in the order: bleached cotton, mercerised and bleached cotton, linen, viscose rayon filament, viscose rayon staple, cuprammonium rayon. W.M.

Mechanism of the Action of Carriers in Dyeing Polyester Fibres with Disperse Dyes

P. Rochas and M. Courmont

Bull. Inst. Text. France (86) (Jan-Feb 1960) 15-40

Twenty-six aromatic compounds effective as carriers were examined in conjunction with 9 disperse dyes. All systems showed the same qualitative behaviour. Up to 50-80 mg. of carrier per g. of fibre can be firmly bound in the amorphous regions of the fibre. This firmly-bound carrier is not removed by water at 80°C. or acetone at 0°C. The water uptake of the fibre is not significantly increased. A further amount of carrier can be weakly bound by the fibre. The dye-uptake increases with increasing amounts of carrier up to the limit of firmly-bound carrier; further carrier is either ineffective or actually reduces dye-uptake. A higher dye-uptake is obtained if carrier and dye are applied in separate baths. For a given dye at a fixed concentration, maximum dye-uptake at optimum carrier uptake is approx. the same for all carriers studied. Increased dye-uptake still persists if the carrier is completely removed and the fibre dried over P_2O_5 . J.C.F.

I-T Process for Applying Cationic Dyes to Acrylic Fibres

J. A. Leddy

Amer. Dyestuff Rep., 49 (18 April 1960) 272-284

The I-T process consists in the use of Irgasol DA (Gy), an anionic agent which forms complexes with cationic dyes, and Tinegal NA (Gy), a non-ionic agent having no appreciable effect on the rate of exhaustion of cationic dyes but which helps to keep the cationic-anionic complexes colloiddally dispersed. Their use results in the dye being largely absorbed below the boil and in dyes which normally have different rates of exhaustion, being absorbed at the same rate. To obtain maximum fastness, however, high temperature dyeing is necessary. Addition of dye can be made without further addition of retarding agent and disperse dyes can be used for final shading. The process can be used for blends of Orlon 42/Dacron 54, Orlon 42/cellulosic fibres, and Orlon 42/nylon. C.O.C.

Dyeing and Finishing Special Blends of Arnel with Acrylics and Arnel with Cotton in Circular Knit Application—III

R. E. Lacy and J. A. Dayvault

Amer. Dyestuff Rep., 49 (7 March 1960) (150)-(152) 37-39

For 50/50 Arnel/Orlon blends neutral dyeing cationic dyes are used for the Orlon and disperse dyes for the Arnel. The dyes are applied simultaneously at pH 6.0-7.5 in presence of Igepon T to promote dye compatibility, retard strike of cationic dyes on to the Orlon and keep the disperse dyes dispersed. The mechanism of the anionic surfactant cationic dye system is one in which a loosely-held surfactant-cationic dye compound is formed. During dyeing the compound dissociates as the concentration of the dye cations decreases and so makes available additional dye cations for the Orlon. Practical recipes and lists of suitable dyes are given. Most colours demanded on Arnel/Acrilan blends can be obtained with disperse dyes only. Some heavy dyeings may need filling in of the Acrilan with cationic dyes. Reserve effects are obtained by dyeing the Acrilan with cationic, acid or premetalised acid dyes and reserving the Arnel. Cross dyed effects are limited as both fibres dye with disperse dyes. Arnel/cotton blends are readily dyed in one bath using disperse dyes for the Arnel and aftertreated direct dyes for the cotton. Blacks are obtained with developed dyes. The dimensional stability given by the Arnel is such that only a cationic softener or a non-resinous anticleur finish is needed. Arnel has no reactive sites for cross-linking with resins which, however, can be used to modify the handle of and stabilise the cotton. C.O.C.

Recent Advances in the Technology of Dyeing Blends containing Dacron Polyester Fibre

P. L. Meunier, R. J. Thomas, J. C. Hoscheit

Amer. Dyestuff Rep., 49 (7 March 1960) (153)-(158) 40-45

Wool/Dacron blends are dyed with acid or mordant dyes for the wool and disperse or cationic dyes for the Dacron. Effect of heat setting before dyeing in the absorption of the dyes by the Dacron is an important consideration especially with lightweight tropical suitings which tend to crease when winch dyed. Data are given

showing the effect on dyeability of heat setting at 280-400°F. for both Type 54 and the new modified Type 64 Dacron. Carrier dyeing of blends containing Dacron is discussed in relation to the acid-modified Type 64 Dacron with disperse and cationic dyes. The effects of dyeing time, dye selection and rates of dyeing on staining of the other components of blends and the resulting effect on fastness to solvent bleeding are emphasised. C.O.C.

Dyeing of Gloving and Garment Leathers

M. Luscher

Rev. tech. ind. cuir, 51 (1959) 223-231

Chem. Abs., 54 (10 March 1960) 5141

Substituted azonaphthol dyes [Derma CW and W(S)] heated with a cationic agent (Dermafix LW) to give difficultly-soluble addition products or with $CuSO_4$ to give copper complexes, give dyeings on gloving and garment leathers of good fastness to light, washing, perspiration, and rubbing. C.O.C.

Dyeing Cellulose Acetate with Disperse Dyes. IX—Effect of Varying Acetyl Value

C. L. Bird and G. Tabbrown

J.S.D.C., 76 (April 1960) 217-222

Reactions of Cold-dyeing Procion Dyes with Cellulose

T. L. Dawson, A. S. Fern, and C. Preston

J.S.D.C., 76 (April 1960) 211-217

PATENTS

Printing and Dyeing Pile Fabrics

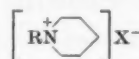
Deep Dye Industries BP 830,927 (U.S.A. 12 May 1955)

The fabric is spread above a number of receptacles having vertical walls with free edges. Pressure is applied to the fabric by a flat rigid plate so that some of the pile enters each receptacle. The walls of each receptacle extend into the pile and so the pile within the receptacle is isolated from the remainder of the pile. Simultaneously a colouring agent is fed into each of the receptacles so that it can be absorbed by the pile in them. C.O.C.

Padding Textiles with Esters of Leuco Vat Dyes

FH BP 833,668 (Germany 2 July 1955)

Adding to the padding liquor a compound of formula—

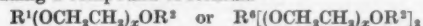


(R = Alk of > 5 C; X = acid residue, e.g. cetylpyridinium chloride, evens up the rates of exhaustion of the different dyes in the liquor. C.O.C.

Padding Vegetable Fibrous Materials with Esters of Leuco Vat Dyes

FH BP 833,674 (Germany 9 July 1955)

Adding a compound of formula—



(R¹ = H, hydrocarbon or acyl; R² = H, hydrocarbon, acyl residue of sulphuric, phosphoric or of a carboxylic or sulphonc acid amide, or -NR³R⁴ or -NR³R⁴R⁵ (R³, R⁴, R⁵ = same or different hydrocarbon and R⁴ and R⁵ may also = H); R¹ and R² cannot simultaneously = H; x = whole number; R³ = divalent hydrocarbon, e.g. condensate of 1 mol. oleic acid with 50 mol. ethylene oxide, evens up the rate of exhaustion on to vegetable fibres of the dyes in the liquor. C.O.C.

Dyeing Nylon or Polyester Fibres

Howards of Ilford BP 831,141 (11 May 1955)

Addition of cyclohexylamides of benzenesulphonic acids, e.g. o-, m- or p-toluenesulphonic acid cyclohexylamide, to disperse dyebaths, increases the proportion of dye taken from the bath by nylon or polyester fibres or the speed of absorption of the dye. C.O.C.

o-Dihydricphenols as Oxidation Dyes for Hair

Monsavon l'Oreal BP 831,851 (France 17 Jan 1958)

Compounds of formula—



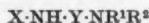
(R = H, Cl, aldehyde, Alk, cycloalkyl, aralkyl, carboxy, carboxyalkyl, alkoxy, nitroalkyl, nitrohydroxyalkyl or amino acid residue) are used as oxidation dyes with NH_4

or alkali metal iodate, periodate or persulphate as the oxidising agent. Thus hair treated with catechol when oxidised with Na iodate becomes a light mahogany or oxidised with K periodate becomes greyish black.

C.O.C.

Dyeing Polyacrylonitrile with Basic or Disperse Dyes with Simultaneous Production of an Antistatic Finish

FBY BP 832,444 (Germany 15 Feb 1957)
Presence in the dyebath of 1–3% on the weight of the polyacrylonitrile of a compound of formula—



(X = acyl residue of a carboxylic or sulphonic acid having a chain of 10–18 C which may be broken by hetero atoms; Y = alkylene of 2–6 C; R¹ and R² = same or different Alk of 1–4 C) or a salt or quaternisation product thereof gives good levelling of basic or disperse dyes. The dyed materials have a very soft handle and excellent antistatic properties.

C.O.C.

Polymeric Amidoximes: Auxiliary Agents: Increasing the Affinity of Acrylonitrile Fibres for Dyes

Gevaert Photo-Producten BP 786,960
Treating a polymer containing nitrile groups with hydroxylamine yields products having many uses, e.g. for adding to paper pulp or impregnating paper, for making into fibres, for improving the affinity for dyes of fibres containing nitrile groups, etc.

BP 833,204

Higher affinity for dyes is obtained if treatment with hydroxylamine is followed by treatment with ammonia or an organic base. Thus treatment with hydroxylamine and then with ammonia or an amine much increases the affinity of acrylonitrile fibres for acid dyes.

C.O.C.

Progress in Leather Dyes (IV p. 455)

Some Relations between the Chemical Constitution of Cibacron Dyes and their Dyeing Characteristics (IV p. 455)

Absorption of Cationic Surfactants by Cellulosic Materials (X p. 463)

Improving the Wet Fastness of Dyeing Leather (XII p. 466)

IX—PRINTING

Structure—Mechanical and Rheological Properties of Aqueous Solutions of Thickening Agents

A. V. Senakhov and F. I. Sadov

Kolloid. zhur., 21 (July–Aug 1959) 476–484

The properties of starch, tragacanth, sodium alginate, fruit gum, sodium carboxymethyl cellulose, and dextrin have been investigated using ordinary technical samples of material. The structure-mechanical properties of soln. of these thickening agents (TA) have been characterised by their elastoplastic-viscous behaviour (P.A. Rebinder *et al.*, *Doklady Akad. Nauk S.S.S.R.*, 71 (1950) 85) using the plate method (*ibid.*, 49 (1945) 354). Study of the kinetics of deformation under stress permits the starch to be distinguished from the remaining TA, the former being characterised by absence of linear regions in the kinetic curves caused by the stiff structural framework of starch. The remaining TA, on the other hand, exhibit linear regions because of their thixotropic properties. Plastic deformation of gels of untreated starch can be achieved only by irreversible breakdown of their internal structure. TA, suitable for use in printing pastes, all have a common structure-mechanical model and similar rheological properties, differing only in the order of magnitude.

G.J.K.

PATENTS

Viscous Oil-in-water Type Printing Pastes

BASF BP 833,602 (Germany 13 Nov 1956)

Oil-in-water emulsions containing a petroleum hydrocarbon in the inner phase and a pigment and a high mol. wt. film-forming material in the outerphase is prevented by addition of a small amount of a poly(ethylene oxide) of mol. wt. 2000–10,000. Addition of an aqueous dye paste to such an emulsion results in marked increase in viscosity and yields a paste highly suitable for blotch printing.

C.O.C.

Extender for Oil-in-water Textile Printing Emulsions

Sherwin-Williams Co. BP 832,809 (U.S.A. 4 April 1956)

A 2–8% aqueous dispersion of a galactomannan produced from seed endosperms of *Leguminosae* is used. The dispersion also contains 0.4–10.0% on the weight of the galactomannan of a ferrous salt and may also contain a hydrocarbon in the dispersed phase.

C.O.C.

Printing on Moving Webs

O. Posch BP 832,453 (Germany 14 May 1957)

A printing roller is loaded with the solid colorants applied to it in the desired pattern. The web to be printed is moistened with a suitable solvent and passed between two squeeze rollers, one of which is driven. The printing roller is driven by it being urged against the web where it lies on the driven squeezing roller. The solvent in the web causes it to pick up colour from the printing roller. The arrangement is such that the length of web which can be seen at once is such that even the largest pattern can be checked. Simple automatic means are provided for changing the printing rollers without them being touched by hand.

C.O.C.

Printing Polyester Fibre Fabrics with Guanidine

FB BP 834,888 (25 July 1955)

The appearance of fabrics containing polyester fibres is altered by printing on them a paste containing a thickening agent and guanidine or a compound yielding guanidine, e.g. guanidine carbonate, and then steaming or heating. Depending upon the length of time and temperature of the steaming or heating the intensity of the effect can be varied, e.g. the printed polyester fibres may become transparent or may be destroyed. Thus a polyester fibre fabric printed with 60 parts of a mixture of British gum (1:1) and crystal gum (1:2) together with water (20 parts) and guanidine carbonate (20), dried, heated for 30 sec. at 200°C., rinsed and soaped at the boil exhibits a printed transparent pattern on a milky ground. Printing a similar fabric with British gum (1:1) (50 parts), guanidine carbonate (25), and water (25), drying, heating for 1 min. at 200°C., rinsing, and soaping at the boil results in the printed parts of the fabric being completely destroyed.

C.O.C.

Multicolour Printing on Hard Foils of Poly(vinyl Chloride)

FB

BP 832,348 (22 Feb 1955)

An ink consisting of a high proportion of a colorant pasted with a binding agent and containing no liquid diluent or, if a drier is added, only the diluent present in the drier can be used for screen-printing of the foils. A very fine screen can be used which results in multi-colour etchings in every way comparable to four-colour prints on paper.

C.O.C.

Photosensitive Composition Developed and Fixed by Heat Alone

Ferro Corpn.

USP 2,905,554 (19 Aug 1957)

A mixture of (a) a halogenated vinyl resin, or a halogen-free vinyl or cellulose resin together with a material highly unstable to light and of b.p. < 150°C., (b) a small amount of ZnO, (c) a small amount of an unsatd. cyclic diketone which may be substituted by Alk and must contain no hydroxyl or amino group, and (d) a small amount of resorcinol monobenzoate, when exposed to light and then heated yields an image having a fastness to light hitherto unobtainable by compositions capable of being developed and fixed by heat alone.

USP 2,905,555 (19 Aug 1957)

A mixture of (a) and (c) together with a little of a Zn compound, and a trivalent cerium compound is used.

USP 2,905,556 (19 Aug 1957)

A mixture of (a), (c), a Zn compound and as a light stabiliser a combination of a trivalent cerium compound and resorcinol monobenzoate, is used.

C.O.C.

Printing and Dyeing File Fabrics (VIII p. 461)

Heat-sensitive Recording Papers (XI p. 466)

X—SIZING AND FINISHING

Masters and Men in the Early Nineteenth Century Finishing Industry

S. H. Higgins

Dyer, 123 (22 April 1960) 649–651

There was distress amongst workpeople after the Napoleonic Wars, and an unsuccessful attempt to start a

Trade Union at Catrine Bleachworks in 1823. In "First Report on Bleaching and Dyeworks, House of Commons 1857", introduction of ten-hour days was opposed by masters of Great Britain. The operatives claimed that even children worked 16 hours a day, and that process re-organisation would give safe and satisfactory work in a ten-hour day. The Act was passed in 1860, and ten-hour day worked well. Relations between unions and employers are now very good. There are references to U.S. bleaching history and practice. R.A.P.

Absorption of Cationic Surfactants by Cellulosic Materials

I—Uptake of Cation and Anion by a Variety of Substrates

F. H. Sexsmith and H. J. White

J. Colloid Sci., 14 (1959) 598-618

Absorption isotherms at 20–35°C. were determined for the cation and anion of cationic surfactants in various aqueous solution-fibre systems at equilibrium. The surfactants were hexadecyltrimethylammonium bromide and octadecyltrimethylammonium bromide, 0.005–100.0 mmoles/l. being used. The fibres used were cotton, medium-high tenacity viscose rayon, viscose rayon monofil and oxycellulose. The major absorption processes were: (1) cation exchange, which depended on the exchange capacity of the substrate, and (2) ion-pair absorption which was important at concentrations near or above the critical concentration for micelle formation. At all except the highest ionic strengths, ion-pair absorption was consistent with the assumption that ions cluster on exchanged long-chain cations. Absorption on oxycellulose of Emcol E-607-L (dodecylxyethylamide of 1-(carboxymethyl)pyridinium chloride) was also studied. The critical concentration for micelle formation of this agent was 0.001–0.002 M. It and the long-chain bromides were much more effective than KBr, NaBr, $(CH_3)_4NBr$ or benzyltrimethylammonium bromide in displacing H^+ ions from oxycellulose. The rates of absorption on the various substrates were consistent with the concept of two different absorption processes. When disperse dyes were added to systems in which the concentration of cationic agent exceeded the critical concentration for micelle formation, the fibres were thoroughly penetrated by dye and the dyeings were fast to washing. Apparently a cationic surfactant is necessary for successful dyeing as similar experiments with Na dodecyl sulphate gave poor results.

II—Effects of Esterification of the Carboxyl Groups in the Cellulosic Substrates

Y. Gotshal, L. Rebenfeld, and H. J. White

Ibid. 619–629

Viscose rayon monofil containing 0.024 millimole of COOH per g. was hydroxylated by exposure for several days to 30–50% aq. soln. of ethylene oxide at 5–25°C. The COOH concentration was reduced to 0.008 millimole per g. Similar experiments with oxycellulose gauze (oxycellulose prepared by NO_2 oxidation) reduced the COOH concentration from 3.45 millimoles/g. at the start to 0.11–1.40 millimoles/g. The capacity of the celluloses to absorb water was little affected by hydroxy-ethylation. Absorption of hexadecyltrimethylammonium bromide was markedly reduced. This is in accord with the decrease in ion-pair absorption to be expected from Sexsmith and White's hypothesis (cf. above) that ion-pair absorption depends on clustering of ions on exchanged long-chain cations.

III—A Theoretical Model for the Absorption Process and Discussion of Maximums in Absorption Curves for Surfactants

F. H. Sexsmith and H. J. White

Ibid. 630–639

Chem. Abs., 54 (10 March 1960) 5097–5098

The model consists of the absorption of cations by an exchange process followed by clustering of ion pairs on the exchanged cations. Application of the law of mass action to the case of a solution of a surfactant capable of forming micelles produces a theoretical relationship of single long-chain ion concentrations to total solute concentration that reproduces the experimental behaviour of aqueous hexadecyltrimethylammonium bromide solutions. The relationship is applicable to cationic or anionic agents. Combining this treatment with considerations of the ion-pair-clustering model provided an equation relating the concentration of micelles to the degree of saturation of

the substrates' capacity for ion-exchange. By inserting reasonable values for the 8 constants, theoretical absorption isotherms were derived resembling in character the experimental isotherms reported in the first two parts. C.O.C.

Formic Acid Colloid of Methylolmelamine Resin Treatment of Cotton: Preliminary Cost Study

O. J. McMillan, K. M. Decossas, W. N. Berard, W. A. Reeves, E. F. Pollard, and E. L. Patton

Amer. Dyestuff Rep., 49 (6) (21 March 1960) 21–32 (180–181)

Data indicating a low cost, under U.S. conditions, for imparting in one treatment resistance to both weather and rotting. C.O.C.

Mechanism of Resin Deposition in Cotton

H. Rath, E. Herbolzheimer, and S. Stapf

Textile Res. J., 30 (March 1960) 201–207

Deposition of synthetic resin in the fibre is independent of the method of application. Thus crease recovery and reduced swelling can be obtained by immersion as well as by spraying the precondensate solution on to the cloth provided suitable equipment is available. As the deposition of resin was practically uniform in all cases during the investigation, the cloths exhibited fairly uniform abrasion resistance whatever method the resin was applied by. While in principle heavy squeezing is not absolutely essential to attain a good crease recovery finish, it is obviously necessary in practice. C.O.C.

Cross-linking of Cotton Cellulose with Quaternary Ammonium Derivatives of Bis Chloromethyl Ethers

G. C. Tesoro

Textile Res. J., 30 (March 1960) 192–201

The cross-linking of cellulose with bis quaternary ammonium compounds derived from bis chloromethyl ethers of aliphatic glycols can be carried out under conditions which minimise or avoid side reactions. Varying the structure of the glycol used enables the effect of cross-link structure on the properties of the cross-linked fibre to be studied. Specifically, effect of cross-link length and rigidity and of hetero atoms and bulk may be investigated and thus information gained about the penetration and reactivity of molecules of varying size and shape. Comparative study of monofunctional compounds, using the pyridinium salts of the chloromethyl ethers prepared from n-butanol, n-octanol, and n-decanol, has been attempted but unfortunately these compounds appear to decompose more readily than bifunctional compounds, and optimum conditions for their reaction with cellulose have not yet been established. 20 references. C.O.C.

Cotton Cross-linked at Various Degrees of Fibre Swelling

W. A. Reeves, R. M. Perkins, and L. H. Chance

Textile Res. J., 30 (March 1960) 179–192

Examination of the wet and dry crease recovery and the wet and dry densities of cloth finished with known types of wash-and-wear finishing agents showed that all the finishes applied by the pad, dry, and bake process had good wet and dry crease recovery. Finishes applied by reaction in aqueous solution produced cloths having increase only in wet crease recovery and these cloths had a more open structure. Examination of these cloths led to study of cloths which had been cross-linked under various states of fibre swelling. Print cloth was cross-linked with HCHO using HCl as catalyst in systems with varying amounts of water and acetic acid. The aqueous systems provided considerable swelling of the fibre before and during cross-linking, while the systems containing large amounts of acetic acid caused relatively little swelling of the fibres. The state of swelling at the time of cross-linking had tremendous influence on fibre structure and on the resulting physical properties of cloth made from the fibres. Wet and dry crease recovery, wet and dry density, affinity for direct dyes, regain, water of imbibition and wash-and-wear properties all differed according to the state of fibre swelling at the time of cross-linking. High degree of swelling resulted in cloth with good wet but little or no increase in dry crease recovery and good drip-dry properties. Relatively little swelling resulted in cloth with good wet and dry wrinkle recovery and also good wash-and-wear properties when either drip dried, line dried or tumble dried. A mechanism, illustrated by diagrams, is proposed for wet and dry crease recovery in cotton cloths.

It embodies three important factors: cross-linking through primary valence cross links, cross-linking through secondary valence bonds, and the position of the cross links. Experimental evidence in support is given. 18 references. C.O.C.

New Development in High Grade Finishes with Special Reference to the Wash-and-Wear Finish—II

H. Enders *Melliand Textilber.*, 40 (Dec 1959) 1454-1462
Measurement of the creasing angle (dry or wet) provides insufficient data to enable conclusions to be drawn regarding wash-and-wear properties. Comparison of different resin finishes, especially with regard to chlorine retention, indicated that epoxy-resins are much superior in this respect. However crease recovery is insufficient. It was found, however, that a combination of ethylenurea, triazone, and epoxy resins gives satisfactory results. A special catalyst must be used for epoxy resins and the use of zinc-fluoroborate and zinc nitrate is discussed. W.M.

Retrospect and Prospect: Progress and Problems in "Wash-and-Wear"—Preliminary Report

P. B. Mack, M. C. Barra, A. L. Muller, E. F. Thomas, and E. F. Herring *Amer. Dyestuff Rep.*, 49 (7) (4 April 1960) 49-62 (228-241)

Study of hand and machine laundering tests made on forty-two types of garments made from cotton, nylon, Dacron, and blends of cotton and Cupion, cotton and nylon, cotton and Dacron, cotton and Arnel, and cotton and Kodol showed that few garments sold as "wash-and-wear" retained such property both as regards appearance of the fabric and of the stitching after a number of launderings. Ironing time was, however, greatly reduced with most garments. Strength losses with few exceptions were negligible after 25 launderings and in most cases dimensional stability was good. As the appearance of the stitching is in many cases a definite cause of poor wash-and-wear performance, studies on seams and other stitching should be made to include consideration of the compatibility of fabric and thread, types of power sewing machine needles and stitches per inch. Strength of common seams with serged edges also needs study, particularly in men's shirts. C.O.C.

Fabrics Impregnated with Urea-Formaldehyde Resins—Effect of Bleaching and Ironing

S. Pierret and R. Bellaton
Bull. Inst. Text. France (86) (Jan-Feb 1960) 137-144

Urea-formaldehyde resin-finished cotton fabric was treated with sodium hypochlorite solution in the presence of variable proportions of non-resin-finished fabric. Chlorine retention by the finished fabric increased with (a) hypochlorite concentration, and (b) the proportion of non-finished fabric present. Liquor ratio had little influence on chlorine retention. Subsequent yellowing during ironing can be prevented by use of an anti-chlor. Sodium bisulphite was found to give good results with short periods of treatment. J.C.F.

Performance Characteristics of Chemically Stabilised Cotton Knit Goods

J. B. Irvine *Textile Res. J.*, 30 (March 1960) 222-233

Chemical stabilisation of cotton knit fabrics reduces shrinkage by half, yields pronounced improvement in the wash-wear appearance, preserves the surface appearance through laundering and imparts crease resistance which helps to maintain better appearance during wearing. The accompanying losses in bursting strength and abrasion resistance have a minimum effect on the use properties of the fabric because of the flexibility of the knitted structure. They are as valuable for use on knit goods as on woven cloths. C.O.C.

Finishing Blends of Orlon and Cotton in Underwear

H. T. Pratt *Amer. Dyestuff Rep.*, 49 (6) (21 March 1960) 43-44 (192-193)

PATENTS

Antistatic Finish

N.V. Onderzoekingsinstituut Research
BP 833,699 (Holland 15 Feb 1956)

Treatment with an aqueous dispersion of a mixture of an organic anionic surfactant and an organic cationic surfactant one of the components being in 30 mol. % excess is used to render hydrophobic fibres antistatic. C.O.C.

Crease Recovery Finish giving Cellulose Fabrics Increased Resistance to Chlorine

Tootal Broadhurst Lee Co. BP 831,297 (17 June 1958)
The fabric is impregnated with a precondensate of 2-5 mol. HCHO, 1 mol. urea and < 1 mol. of glyoxal and baked in presence of an acid catalyst. C.O.C.

Improving the Properties of Cellulosic Fibrous Sheets by Treatment with Alkali

Carl Freudenberg BP 830,904 (Germany 20 Aug 1955)

Rapid welding together of cellulosic fibres is obtained by treating them with alkali liquors containing a swelling agent, e.g. a zincate, stannate, beryllate or an iron-tartaric acid-alkali complex, e.g. $[(C_6H_5O_4)Fe]Na_3$ (Jayme and Verburg, *Rayon, Zellwolle und andere Chemiefasern*, 32 (1954) 193, 275) subjecting them to pressure and washing free from alkali. C.O.C.

Gas-plating Heat-sensitive Filaments and Fibres

Commonwealth Engineering Co. of Ohio

BP 830,635 (7 March 1958)

The fibres are coated with a heat-resistant flexible resin and then treated with a gaseous metal compound at a temperature sufficient to cause thermal decomposition of the metal compound and consequent deposition of metal on the fibres. C.O.C.

Crêpe Fabric of Synthetic Fibres

Toyo Rayon and Sakai Sen-I Kogyo

BP 833,491 (Japan 11 May 1955)

Synthetic fibre yarn is heat set after being given a high degree of twist. It is then twisted in the opposite direction beyond the zero point of twist, sized, and dried to prevent shrinking and crimping. It is then used either as weft or as warp and weft in a fabric which is then washed with agitation at < 70°C. This causes the fabric to shrink 30-40%. It is then again washed at a higher temperature to desize it completely. Finally it is set at the desired width by steam and/or dry heat. C.O.C.

Bonding Synthetic High Polymers to Polyurethane Elastomers

General Tire & Rubber Co. BP 833,075 (27 July 1956)

Nylon or polyester fibres coated with the infusible, insoluble resinous polyhydricphenol-aldehyde condensate have excellent adhesion to polyurethane elastomers applied to them under pressure. C.O.C.

Applying Resilient Rubbery Backings

Lintafoam BP 833,476 (23 Jan 1957)

Crumb rubber is mixed with an unstable foamed rubber latex compound and continuously fed and spread into a thin layer while supported by a moving belt. This layer is formed on the material to be backed or the material to be backed is passed into the layer which has been formed directly on the belt. The layer and the material to be backed are held together on the belt at a moderate temperature until the foamed latex collapses and gels after which the latex is vulcanised. C.O.C.

Amidophosphoric Acid Derivative—Flame Resistant Finishes (III p. 451)

Dyeing and Finishing Special Blends of Arnel with Acrylics and Arnel with Cotton in Circular Knit Application—III (VIII p. 461)

Dyeing Polyacrylonitrile with Basic or Disperse Dyes with Simultaneous Production of an Antistatic Finish (VIII p. 462)

Oxidative Degradation of Cellulose in Alkaline Solutions (XI p. 465)

Reinforced Paper (XI p. 466)

Flexible Material Made Up of Several Layers (XI p. 466)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Properties and Nature of the Surface of Cellulose—I and II

K. Borgan *Norsk Skogind.*, 13 (1959) 81-92, 429-442
Chem. Abs., 54 (10 March 1960) 5081

Apparatus for determining the contact angle between different liquids and cellulose is described. It makes possible determination of the contact angle of cellulose at any R.H. between 0-100% and at 0-100°C. Different cellulose specimens were prepared by bringing the sample into solution and then precipitating it as a film. Pure cellulose films were made by deacetylating cellulose acetate

and by hydrolytic coagulation of cellulose xanthate. The contact angle of the system water-cellulose-air is a function of the water content of the cellulose, the R.H. of the air, temperature, and time. It is nearly independent of R.H. at 0-50% but as R.H. increases above 50% the contact angle decreases. Thus cellulose can absorb up to 12% water without apparent changes in the properties of its surface. Up to this moisture content the water is present as a true hydrate whereas above 12% it is so weakly combined with the cellulose that the number of free accessible OH groups on the system cellulose-water increases. The hydrophilic character of cellulose can be characterised by the contact angle of water-cellulose-air, which was 10-2-18-7° when the R.H. varied from 0-100%. Thus cellulose is not so hydrophilic as would be expected from its chemical constitution. This is probably because the number of free hydroxyl groups present is less than normally assumed. Use of a special microtechnique showed that because of diffusion of water into cellulose and formation of hydrates, the hydrophilic properties of cellulose are not constant until an equilibrium has been reached. This is the real reason for the spreading of water on the surface of cellulose and for the fact that the contact angle decreases with time.

C.O.C.

Specific Surface Area of Cellulose

K. P. Mishchenko, S. L. Talmud, and V. I. Yakimova
Kolloid. zhur., 21 (May-June 1959) 330-335

Nitrogen adsorption and ion exchange methods furnish reliable values for the specific surface area (SSA) of cellulose. The corresponding values for bleached cotton cellulose are 18-94 (adsorption) and 16-74 sq.m./g. (ion exchange); swelling in water apparently not affecting SSA. On the other hand, celluloses from wood pulp, whilst exhibiting a much smaller degree of porosity in the dry state (SSA for bleached sulphite wood pulp is 2-23 sq.m./g.), increase their SSA considerably on swelling (100-200 sq.m./g.). In the ion exchange method 0.1 N-CaCl₂ is used (pH 1-6).

G.J.K.

Homogenisation of Cellulose with Respect to Molecular Weight by Activated Oxidation

B. A. Zakharov, V. I. Ivanov, and G. A. Krýlova
Doklady Akad. Nauk S.S.S.R., 127 (11 July 1959) 396-397

Partially bleached and scoured sulphate cellulose, which has been pretreated ("activated") with urea, undergoes considerable homogenisation on bleaching with hypochlorite (pH 11); the distribution of mol.wt. curve showing a mean max. for DP 800. It is assumed that activation with urea renders the long-chain molecules more readily accessible to the action of oxidising agents, resulting in "specific" degradation.

G.J.K.

Oxidative Degradation of Cellulose in Alkaline Solutions

H. Vink
Arkiv. kemi., 14 (1959) 195-209 (in English)
Chem. Abs., 54 (10 March 1960) 5081

Study of the kinetics of the oxidative degradation of cellulose in alkaline solutions with special reference to the possible presence of weak links in the cellulose. Viscosity measurements were made of various celluloses in cupriethylenediamine solutions. Certain anomalies in the degradation were noticed and found to be caused by formation of inhibitors in the solutions than by weak links in the cellulose. The inhibitors were isolated and identified as oximes produced by oxidation of the ethylene diamine. Quantitative studies of the effect of these and other oximes on the degradation indicated that the degradation is a random process and no evidence for weak links was found.

C.O.C.

Distribution of Substituents in Cellulose Ethers

I. Croon
Svensk Papperstidning, 63 (30 April 1960) 247-256 (in English)
C.O.C.

Literature review, 42 references.

Preparation of β -1,6-Anhydro-1,5-Glucopyranose (Levogluconan) by Thermal Decomposition of Cellulose with Steam under reduced Pressure

Ya. V. Epshtein, O. P. Golova, and L. U. Duryina
Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk., No. 6 (June 1959) 1126-1127

Thermal decompn. of cotton cellulose in a stream of superheated steam, or inert gases, at 24-30 mm. Hg, leads to the formation of levogluconan in 40% yield.

G.J.K.

Infrared Spectra of Cellulose Modifications

R. T. O'Connor, E. R. McCall, and D. Mitcham
Amer. Dyestuff Rep., 49 (7) (4 April 1960) 35-43 (214-222)

A review of the literature, 37 references.

C.O.C.

Constitution of the Hemicelluloses of Sitka Spruce. III—Structure of an Arabomethoxyglucuronoxylan

G. G. S. Dutton and K. Hunt
J. Amer. Chem. Soc., 82 (5 April 1960) 1682-1684

Sitka spruce hemicelluloses were methylated after extraction with 5% KOH of a chlorite preparation and yielded a homogeneous sample after fractional pptn. 2,3,4-tri-O-methyl-D-xylose (1 mole), 2,3,5-tri-O-methyl-L-arabinose (0.4 mole), 2,3-di-O-methyl-D-xylose (10 moles), 2-O-methyl-D-xylose (0.3 mole), 3-O-methyl-D-xylose (1.2 moles), and 2-O-(2,3,4-tri-O-methyl-D-glucuronosyl)-3-O-methyl-D-xylose (3.4 moles) were obtained on hydrolysis. Structural features of the polysaccharide are discussed, the main feature of the hemicellulose being a chain of D-xylopyranose units linked through positions 1 and 4.

F.J.

Hydrolytic Hydrogenation of Cellulose

A. A. Balandin, N. A. Vasyunina, S. V. Chepigo, and G. S. Barýsheva
Doklady Akad. Nauk S.S.S.R., 128 (11 Oct 1959) 941-944

Hydrolytic hydrogenation of cellulose in presence of dil. H₃PO₄ and Ru (catalyst) in a rotating autoclave yields sorbitol (92-96% from sulphite and 85-90% from cotton CE). Optimum conditions are: 160-165°C., 60-80 atm. H₂, 50-60 min., 0.7% H₃PO₄. The superiority of Ru over other transition metal catalysts is explained in terms of ionic radius of catalyst to length of catalysed bond (C=O) relation.

G.J.K.

The Alkaline Degradation of Chemically Modified Cellulose

W. M. Corbett
J.S.D.C., 76 (May 1960) 265-271

PATENTS

Colouring of Artificial Cellular Products

Sponcel
BP 833,212 (18 July 1955)
Coloured cellular regenerated-cellulose products are obtained if coloured fibres are incorporated in the cellulose xanthate used as the raw material.

C.O.C.

Chemically-reactive Allylcellulose

Regie Nationale des Usines Renault
BP 831,969 (France 18 June 1955)

Cellulose treated with 10-30% aq. alkaline metal hydroxide at < 40°C. and then with an allyl halide in an inert solvent, e.g. CCl₄, at < 40°C. in an inert atmosphere, e.g. N, does not lose its fibrous structure. The product contains reactive OH groups and double bonds.

C.O.C.

High-speed Coating of Paper

Kimberly-Clark Corporation
BP 832,073 (23 Oct 1956)

High-quality coatings of improved brightness, opacity, and bulk at low supercalendering pressures and high coating speeds are obtained by application of a setting agent, e.g. alum, to the coated web by a rotating pressure roll while the web is still wet. The time of contact is extremely short, but sufficient to initiate the reaction between the setting agent and the binder, e.g. soya protein, carboxymethyl cellulose, styrene/butadiene copolymer, sodium alginate, styrene/maleic anhydride copolymer.

R.A.

Pressure-markable Coated Sheets

Varco
BP 832,707 (U.S.A. 3 Aug 1956)

The coating consists of solid particles of dark coloured matter individually coated with a pigment of light colour. On being pressed the particles are crushed and the dark-coloured matter exposed. Thus petroleum base pitch of particle size 20-100 μ . is stirred in a bath of wax and solvent, the waxed pigment particles filtered off and the solvent removed from them. They are then treated with TiO₂ smoke (formed from TiCl₄ vapour in hot humid air) so as to completely coat each particle. They are then applied to paper whose surface has been coated with a latex adhesive.

C.O.C.

Resin-filled Paper

BASF
BP 834,757 (Germany 27 July 1957)

Addition of 5-80% of a porous, hardened phenol- or urea-formaldehyde condensate to the pulp improves the

bulk, scuff resistance, surface smoothness, and resistance to combustion of the resultant paper or board. R.A.

Reducing the Tendency of Cast-coated Paper to Curl

S. D. Warren Co. BP 837,072 (U.S.A. 3 June 1957)
The wet coated side of the paper web is pressed against the surface of a moving drum by a pressure roller which has a soft, elastic, stretchable layer and an outer non-stretchable covering. If the pressure-nip between the drum and the roller is sufficiently wide, the paper will not be stretched > 0.25% and the resulting sheet will show very little tendency to curl. R.A.

Reinforced Paper

Mosinee Paper Mills Co. BP 834,850 (U.S.A. 31 Dec 1956)
A leno-weave rayon or nylon scrim is coated on both sides with 1–20% of a synthetic rubber or vinyl polymer composition and fed to the pulp suspension in the head box of the paper machine whence it passes through the slice on to the wire in the usual way. R.A.

Drying Paper Webs

Wiggins, Teape & Co. BP 835,238 (9 Dec 1955)
From the presses the web is passed to a stentering machine, of the type used in the cotton finishing industry, and held under tension by its edges during drying to prevent or limit planar distortion. R.A.

Heat-sensitive Recording Papers

Fritz Hellige & Co. BP 834,184 (Germany 8 Aug 1957)
Coloured paper coated with cellulose acetobutyrate, m.p. 185–240°C., and a plasticiser dissolved in a volatile solvent. This gives a white coating which when pressed with, e.g. a hot metal style, melts to reveal the coloured paper. C.O.C.

Flexible Material Made Up of Several Layers

Kodak BP 834,525–8 (U.S.A. 23 Feb 1955)
A flexible web is given a number of successive coatings. Each coating composition before it is applied to the web is formed into a flowing layer of uniform thickness and of length in the direction of flow many times greater than its thickness. All the layers are simultaneously fed from a coating device above the surface of the web so as to form a coating bead. The feeding is such that all the layers are brought into contact with one another either in the coating bead or before reaching it. This coating bead bridges the gap between the coating device and the web. C.O.C.

Polymeric Amidoximes: Auxiliary Agents: Increasing the Affinity of Acrylonitrile Fibres for Dyes (VIII p. 462)
Absorption of Cationic Surfactants by Cellulosic Materials (X p. 463)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

States of Aggregation of High-molecular Compounds IV—Collagen, Gelatine, and Silk Fibroin

R. I. Fel'dman

Kolloid. zhur., 21 (July–Aug 1959) 499–506
The method of linear dilatometry already developed (*Kolloid. zhur.*, 20 (1958) 220) can be employed successfully when studying the behaviour of fibrous proteins, e.g. temp. dependent changes of length and wt. for collagen (1–252.5°C.), gelatine (15–90°C.), and silk fibroin (2–225°C.); for the latter also under varying conditions of humidity. Of the complex factors affecting linear stretching and contraction of protein an important rôle is assigned to changes in moisture content. In fibrous proteins part of the adsorbed water is less strongly bonded than in polyamides. Changes in the system around 40°C., as shown in dilatometric curves, are connected with very high mobility on the part of some adsorbed water and confirm presence of hydrogen bonding. A different, more intense bonding is indicated by transition points near 100°C.; changes in dilatometric curves near 190°C. may be caused by the protein component becoming highly elastic. It is emphasised that the point of softening of dry collagen (approx. 240°C.), and of silk fibroin, is close to the analogous point for synthetic polyamides. Changes in length are given in relative units rendering comparison on an absolute scale impossible. G.J.K.

Quinone Tannage

R. Lasserre *Bull. assoc. franç. chim. inds. cuir et doc. sci. et tech. inds. cuir*, 21 (1959) 222–230
Review, 33 references. C.O.C.

Precipitation of Vegetable Tannins by Formaldehyde and Urea

R. Lasserre *Bull. assoc. franç. chim. inds. cuir et doc. sci. et tech. inds. cuir*, 21 (1959) 148–161

Staining of Materials in the Skin with Fluorescent Dyes

P. Stadler and W. Träsch *Leder*, 10 (1959) 213
J. Soc. Leather Tr. Chem., 44 (May 1960) 247
Describes apparatus for observing treated skin sections. Actions of 23 dyes on various skin components are tabulated. C.O.C.

PATENTS

Filling of Leather

FBY BP 832,664 (Germany 17 May 1957)
The leather is treated with an aqueous dispersion of fillers in presence, as dispersing agent, of the condensate obtained from a carbamide and formaldehyde in a non-acidic medium containing a water-soluble salt of an organic sulphonic acid. Treatment is completed by bringing the bath to pH 3.5–5.5. C.O.C.

Dehydrating Skins with Organic Solvent and then Vegetable Tanning Them

A. E. Ushakoff USP 2,906,591 (21 June 1954)
The skins are dehydrated by treatment with an organic solvent and then impregnated with an organic solvent solution of a vegetable tannin. Finally they are acidified and dried. Preferably the skins and the tannin solution have pH < 2.0 and the tannin is caused to deposit in the skins by bringing the pH to > 6.0 before the final acidification. C.O.C.

Improving the Wet Fastness of Dyeing Leather

General Aniline USP 2,908,543
The dyed leather is treated with an acidic aqueous solution of the product obtained by heating 1–3 mol. HCHO, 0.5–1.0 mol. NH₄ salt of a strong acid and 1 mol. dicyandiamide at < 45°C. in acidic aqueous solution. This greatly improves the wet fastness properties of the dyeings. C.O.C.

Water-repellent Organopolysiloxane Compositions for Leather

General Electric Co. BP 834,088 (U.S.A. 28 Sept 1956)
A water-repellent composition which cures instantaneously when applied to leather and has no tendency to migrate comprises by weight (1) 9–19% of the intercondensate of (a) a cohydrolysis product containing Si-bonded OH groups of a trialkylsilane and an alkyl silicate and (b) a linear high viscosity organopolysiloxane liquid containing terminal Si-bonded OH groups, (2) 9–19% of a hydrolysed alkylsilane containing a curing agent, (3) 24–34% of a compound of formula (RO)₃Ti (R = Alk of < 13 C), and (4) 38–48% of a solvent. C.O.C.

Progress in Leather Dyes (IV p. 455)

New Method of Preparing Pigments for Leather Finishes (IV p. 455)

XIII—RUBBER; RESINS; PLASTICS

Radiation-induced Copolymerisation of Acrylonitrile with Propylene

L. N. Andreev, B. A. Krentsel', A. D. Litmanovich, L. S. Polak, and A. V. Topchiev
Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, No. 8 (Aug 1959) 1507

Mixtures of acrylonitrile and propylene were irradiated (γ -Co⁶⁰; dose power 200 r per sec.) in an autoclave (room temp.) and the products extracted with dimethylformamide, n-heptane, and ether. On changing the wt. ratio of acrylonitrile to propylene from 0.75 to 0.15 the proportion of propylene links in the copolymer decreased from 26 to 12%. G.J.K.

Polymer Viscoelasticity and Liquid Structure

P. Mason *Research*, 13 (June 1960) 234–240
Many different mechanical properties of polymers can be measured under different boundary conditions but all can be expressed in terms of a distribution of relaxation

times. These properties are strikingly dependent on both time and temperature. Quantitative equivalence between the effect of time and temperature has been established and theoretical treatments emphasise the liquid-like character of the processes involved. C.O.C.

Orientation of Massive Polymeric Materials

M. M. Gudimov, V. A. Kargin, B. V. Petrov, and M. V. Dumnov

Doklady Akad. Nauk S.S.S.R., 128 (1 Oct 1959) 715-718

Study of the effect of induced orientation in massive (linear) polymers, e.g. polymethylmetacrylate, on their physico-mechanical properties. Orientation was affected either by stretching or compression, in the latter case the polymer being in a highly elastic state. A formula for the degree of stretching (E_p) or compression (E_p) respectively, is given: $E_p = (\sqrt{T_1/T_2} - 1) \times 100$, T_1 and T_2 = initial and final thickness of the polymer respectively. For identical E_p and E_p the physico-mechanical indices of the polymer are found to be practically identical. Stretching above 50-70% does not improve the properties of the polymer. Higher degree of molecular order in the polymer results in faster flow of relaxational processes; the polymer becoming, e.g. less sensitive to stress concentrators. G.J.K.

Effect of Pressure on the Ability of Nuclear-substituted α -Methylstyrenes to Undergo Polymerisation I—Polymerisation of para Substituted α -Methylstyrenes

V. V. Korshak, A. M. Polyakova, and I. M. Stoletova
Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, No. 8 (Aug 1959) 1471-1476

p-a-Dimethyl-, *p*-chloro- α -methyl-, and *p*-bromo- α -methyl styrenes undergo polymerisation under pressure (6,000 atm; 120°C.; 5 hr.) in absence of an initiator, the rate of reaction and mol.wt. of the polymer increasing with increase in pressure. Data for the preparation of the monomers and for the thermo-mechanical properties of the polymers are given.

II—Ortho Substituted α -Methylstyrenes

Ibid., 1477-1479
Despite presence of an organic peroxide, *o*-chloro- α -methyl-, 2,5-dichloro- α -methyl-, and *o*- α -dimethylstyrenes do not polymerise at 6,000 atm. and 120-180°C. in absence of initiators because of steric hindrance, but they do form, under these conditions, copolymers with styrene and *p*-chlorostyrene which are soluble in benzene and its homologues. Specific viscosity in toluene (at 20°C.) and thermo-mechanical properties of these copolymers have been determined. G.J.K.

PATENTS

Mottled Continuous Sheet Material

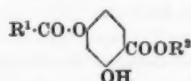
Armstrong Cork Co. USP 2,908,042

Device for feeding granular colour composition to a sheeting calender so that sufficient material is supplied to form a solid unitary sheet without excess to cause extrusion. C.O.C.

β -Resorcylic Acid Diesters for Stabilising Hydrocarbon Polymers to Ultraviolet Radiation

Dow Chemical Co. USP 2,910,454

Compounds of formula—



(R^1 = Ar, halogenophenyl, hydroxyphenyl and Alk of 1-7 C; R^2 = Alk of 1-8 C or subst. or unsubst. Ar) are used, e.g. phenyl 4-benzoxysalicylate gives excellent protection to polyethylene. C.O.C.

Making Polyethylene Film Receptive to Organic Coatings

DuP USP 2,909,443

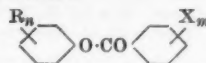
Embedding solid particles into the surface of thermoplastic film causes surface roughening which greatly improves the printability of the film and its adhesion to polymeric coatings, etc. The film's transparency is unaffected as long as the particles are no greater than half the thickness of the film and in no case exceed 50 μ . C.O.C.

Stabilising Halogenoethylene Polymers to Light

Dow Chemical Co.

USP 2,910,453

Compounds of formula—



(R = Alk; X = H, Hal or Alk; m and n = whole numbers) e.g. 4-*t*-butylphenyl-2,4-dichlorobenzoate, give excellent stability to light to halogenoethylene polymers. C.O.C.

Dispersing Pigments or Resins in Polystyrene using Boric Acid

Monsanto Chemical Co.

USP 2,910,450

When extruding styrene polymer particles into strands which are chopped into pellets for use in the moulding trade much more uniform dispersion of pigments, resins, etc. added to the polystyrene in the extruder barrel, and a smooth steady rate of extrusion are obtained if 0.02-2.0 parts of boric acid are added to each 100 parts of polystyrene. C.O.C.

Thermoplastic Moulding Composition comprising Resin Particles coated with Pigment

DuP

BP 835,363 (U.S.A. 29 March 1957)

Granules of thermoplastic resin are coated with 0.5-2.0% by weight of a finely-divided, heat-stable pigment and 0.05-0.5% of a polysiloxane of b.p. higher than the m.p. of the resin. Thus nylon "naturalflake" is treated with 0.2% of the silicone oil DC-200 (Dow Corning) and then with 0.5% of Cadmium Yellow (C.I. Pigment Yellow 37). C.O.C.

Laminated Sheets

ICI

BP 834,454 (11 Jan 1957)

A sheet of poly(vinyl chloride) and a sheet of foamed polyurethane are bonded together by a suitable adhesive. Each sheet is independently embossed with the same pattern and they are bonded together so that the patterns are superimposed one upon the other. C.O.C.

Linoleum Compositions

Armstrong Cork Co. BP 834,845 (U.S.A. 25 June 1956)

A linoleum composition of improved rate of curing and colour comprises a linoleum cement containing oxidised drying oil and resin and 1-15% (on wt. of oil and resin) of a monomeric aliphatic hydrocarbon-substituted polymethylol (or polyethylol) phenol. C.O.C.

Laminating or Coating of Fluoropolyethylenes with or on other Substances

Connecticut Hard Rubber Co.

BP 834,170 (U.S.A. 18 April 1958)

Modification of BP 737,286 and 790,018. Adhesion of the fluoropolyethylene is obtained by fusing it to the surface with an adhesive-receptive synthetic or natural polymer or resin. Thus polytetrafluoroethylene film treated with a methylethylketone solution of Epon 828 (Shell) (an epoxy resin) and baked at 770°F. for 10 min. results in the film and the coating being fused together. Adhesive substances readily and securely adhere to the coated film. C.O.C.

Incorporating Pigment into Polyethylene

General Aniline

BP 834,160 (U.S.A. 10 April 1957)

Addition of a little *N*-vinyl- α -pyrrolidone polymer together with the pigment, either organic or inorganic, results in rapid and even dispersion of the pigment throughout the polyethylene with no tendency for streakiness or mottling. C.O.C.

Flexible Material Made Up of Several Layers (XI p. 466)

XIV—ANALYSIS; TESTING; APPARATUS

Colorimetric Determination of Sulphate in Water Using Ferrithiocyanate

A. K. Balko and L. V. Markova

Ukrain. khim. zhur., 25, No. 4 (1959) 505-508

Optical d of ferrithiocyanate soln. decreases in proportion to increasing sulphate ion concn. Standard soln. contains 10^{-3} M-Fe(NO₃)₃, 10^{-3} M-NH₄CNS, pH 2.7 (Na₂CO₃ and sulphuric acid); light filter having $\lambda = 420$ m μ . Lower limit is 1.2 μ g. SO₄²⁻ per ml. Fluoride and excess ferric ions interfere. Good agreement with gravimetric sulphate determination is claimed. G.J.K.

***R_f* Values of Some Naphthols and Related Compounds**

J. Chromatography, 3 (May 1960) D33
R_f values of some naphthols and similar compounds tabulated from original data by R. J. W. Byrde, D. F. Downing, and D. Woodcock, *J. Biochem.*, 72 (1959) 344. F.J.

Detection of Diphenyl and Its Derivatives

F. Feigl and D. Goldstein
Anal. Chem., 32 (June 1960) 861-862
 Fusion of diphenyl and its derivatives with hydrated oxalic acid at 160-190°C. gives a blue colour within several minutes. A positive reaction is also given by carbazole derivatives. P.B.S.

The Separation and Identification of Commercial Dyes by Paper Chromatography. I—Acid Dyes

C. McNeil *J.S.D.C.*, 76 (May 1960) 272-275

Determination of the Solubility of Dyes

M. Capponi *Tintex*, 25 (April 1960) 235-241
 A method for determining true solubility is described. It depends on the assumption that when solutions of different concentrations are filtered through a suitable filter there will be a linear relationship between the amount of dye retained by the filter and the concentration of the filtered solution as long as the solubility is not exceeded. As soon as the maximum true solubility is exceeded this relationship will suddenly steeply rise. Details of the method and a list of solubilities of a number of Sandoz dyes are given. W.M.

Identification of Flavanone Aglycones by Paper Chromatography of Their Degradation Products

W. J. Dunlap and S. H. Wender
J. Chromatography, 3 (May 1960) 505-507
 Alkaline (30% KOH) degradation of flavanone aglycones such as hesperetin, homoeiridictyol and isoakuranetan, found in citrus fruits, yielded phloroglucinol and substituted cinnamic acids. *R_f* values in different solvents, fluorescence and colour reactions of degradation products enable positive identification and identity of original molecule to be established with certainty. F.J.

Detection of Xanthophyll used as a Food Dye in Pastes

M. Brogioni and V. Franconi
Chimica (Milan), 35 (1959) 540-547
Chem. Abs., 54 (10 April 1960) 6985
 The finely powdered paste is put in a cylinder and eluted with petroleum ether (30-35 c.c./25 g. paste). The solution is concentrated to 1 c.c. under reduced pressure and, without heating, put on top of a column of 6-10 g. activated alumina and eluted with petroleum ether. The column is cut into several portions which are extracted with butanol saturated with water, the extracts filtered and examined spectrophotometrically. Presence of carotenol or xanthophyll epoxide indicates presence of xanthophyll as a colorant in the sample; β -carotene does not interfere. C.O.C.

Determination of Some Dyes for Liquors

R. De Gori, F. Grandi, and F. Santucci
Boll. lab. chim. provinciali (Bologna), 10 (1959) 248-255
Chem. Abs., 54 (10 April 1960) 7059
 In Italy use of Patent Blue V (C.I. Food Blue 3) is permitted in combinations with Indigo Carmine (C.I. Food Blue 1), Brilliant Black BN (C.I. Food Black 1), Sunset Yellow FCF (C.I. Food Yellow 3), Quinoline Yellow (C.I. Food Yellow 13) and Tartrazine (C.I. Food Yellow 4). Spectrophotometric methods have been developed for determining these dyes in solution without separating them. The two closest absorption bands (for Tartrazine and Quinoline Yellow) are distinct enough to enable presence of both to be determined. C.O.C.

Comparison Between Two Methods of Measuring the Flammability of Fabrics

H. G. Wraight and P. H. Thomas
J. Text. Inst., 51 (May 1960) T 203-T 207
 Rates of spread of flame up fabrics both vertically and at 45° are given and the relationship between the two

methods of test making use of these different orientations of the fabric is discussed. C.O.C.

Testing Resistance to Microbiological Rotting

R. Ashcroft and W. A. Bell
J. Textile Inst., 51 (May 1960) T 175-T 189
 A modified soil burial test in which strips of cloth are laid across bands of soil ($\frac{1}{2}$ in. thick) on glass plates and incubated under controlled temperature, humidity, and air supply. The testing arrangement is compact and the samples are visible throughout the test. Experiments to assess the uniformity of the biological conditions and to examine the effect of variations in the temperature, moisture conditions, and the composition of the inoculation are described. C.O.C.

Behaviour of Pleated Strips of Wool Fabric in an Extensometer

H. W. Holdaway
Text. Research J., 30 (April 1960) 296-304
 Idealised load-extension characteristics taken on pleat samples behave to a first order in the manner predicted for an ideal pleated elastica. There was linear relationship between both extension and lateral deflection and reciprocal square root of load. These relationships were valid for tensions 0.75-16 g./cm. width of fabric used and also for various lightweight tropical worsteds. C.O.C.

Practical Identification of Some Condensed Tannins in Leather by Chromatographic and Photometric Methods

D. G. Roux *J. Soc. Amer. Leather Chem. Assoc.*, 55 (May 1960) 272-284
 Methods for identifying some condensed tannins present in leather are based on use of conventional paper chromatography coupled with selective spray reagents and on generation of anthocyanidins from condensed tannins. The anthocyanidins may be recognised by their colour, *R_f* values and light-absorption characteristics. Some practical examples are given. The usefulness and the limitations of the methods are evaluated. C.O.C.

Measurement of Wet-rub Resistance of Paper Coatings

G. A. Hull
Paper Ind., 41 (1960) 782-783
Chem. Abs., 54 (10 April 1960) 7146
 Apparatus which removes the coating uniformly is described. In a typical series of 24 tests on paper containing 25% by wt. of coating, the amount removed in each test was 13.6% with a standard deviation of $\pm 0.9\%$. C.O.C.

PATENT

Measuring the Viscosity of a Coating Liquid

Chemical Products Corp.
BP 833,943 (U.S.A. 29 Jan 1957)
 A viscosimeter comprises a cylinder having openings at each end. This is filled with the coating liquor and yarn or wire drawn through the openings and the liquor. The force necessary to pull the yarn through the liquor is a measure of liquor viscosity. It is particularly suited for testing the coating properties of liquors to be applied to yarn or wire as the actual yarn or wire to be coated can be used for making the test. C.O.C.

Infrared Spectra of Cellulose Modifications (XI p. 465)

XV— MISCELLANEOUS

Manufacture of Coloured Concrete Tiles by use of Inorganic Pigments

R. G. Peasch *Betonstein Zeitung*, 25 (1959) 413-419

PATENT

Anodising Aluminium and Its Alloys

ICI *BP 834,714 (11 Sept 1957)*
 Anodising is accomplished by use of A.C. of frequency 100-500,000 cycles per sec., the electrolyte being preferably at 50-70°C. This yields a tough coating which is more flexible than those produced hitherto. C.O.C.

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